No. 14-1646

UNITED STATES COURT OF APPEALS FOR THE FEDERAL CIRCUIT

TRANSWEB, LLC,

Plaintiff-Appellee,

v.

3M INNOVATIVE PROPERTIES COMPANY AND 3M COMPANY,

Defendants-Appellants.

On Appeal from the United States District Court
for the District of New Jersey in Case No. 10-cv-04413, Judge Faith S. Hochberg

BRIEF FOR APPELLANTS 3M INNOVATIVE PROPERTIES COMPANY AND 3M COMPANY

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CERTIFICATE OF INTEREST

Counsel for Defendants-Appellants certifies the following:

1. The full name of every party or amicus represented by us is:

   3M Company and 3M Innovative Properties Company

2. The name of the real party in interest (if the party named in the caption is not the real party in interest) represented by us is:

   Not applicable

3. All parent corporations and any publicly held companies that own 10 percent or more of the stock of the party or amicus curiae represented by us are:

   There are no parent corporations or publicly held companies that own 10 percent or more of the stock of 3M Company.

   3M Financial Management Company owns 100 percent of the stock of 3M Innovative Properties Company; 3M Company owns 100 percent of the stock of 3M Financial Management Company.

4. The names of all law firms and the partners or associates who appeared for the party or amicus now represented by us in the trial court or agency or have or are expected to appear in this Court are:

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/s/ Seth P. Waxman
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November 3, 2014

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STATEMENT OF RELATED CASES

No other appeals from the district court action presently on appeal were previously before this or any other appellate court. Counsel are unaware of any cases that will directly affect or be directly affected by this Court's decision in the present appeal.

JURISDICTIONAL STATEMENT

The district court had jurisdiction under 28 U.S.C. §§ 1331 and 1338(a). This Court has jurisdiction under 28 U.S.C. § 1295(a)(1). This appeal was timely noticed on July 9, 2014.

ISSUES ON APPEAL

1. Should the invalidity, inequitable conduct, and *Walker Process* antitrust judgments be reversed given that TransWeb's public use argument depended entirely on its own president's uncorroborated assertion that he displayed plasma-fluorinated respirator media at a trade show fifteen years earlier?

This first issue requires reversal of the entire judgment and remand to address 3M's unadjudicated motion for JMOL of infringement. To the extent the Court decides to go further, the other issues on appeal are as follows:

2. Should the judgment of invalidity be reversed given that, in addition to the failure of TransWeb's public use argument (issue 1), the district court failed to consider whether it would have been obvious to make the leap from TransWeb's

alleged public use to 3M's significantly-improved patented method of making filter media?

3. Should the judgment of inequitable conduct be reversed given that, in addition to the failure of TransWeb's public use argument (issue 1), (a) there was not clear and convincing evidence that the inventor and prosecuting attorney knew there had been any alleged public use at the trade show, (b) intent to deceive was not the single most reasonable inference, and (c) TransWeb admitted that the two other items that the court arguably relied on could not have supported a finding of public use?

4. Should the *Walker Process* antitrust judgment be reversed given that, in addition to the failures of TransWeb's public use and inequitable conduct arguments (issues 1 and 3), (a) TransWeb artificially narrowed its market definition to exclude competition from other filter media and foreign respirator manufacturers, and (b) almost all of the $26 million judgment consisted of TransWeb's trebled attorney's fees without any showing of a link between those fees and any actual or threatened harm to competition?

5. At a minimum, is 3M entitled to a new trial?

INTRODUCTION

The district court relied on an accused infringer's newly-minted, uncorroborated allegations regarding the alleged distribution of samples at a trade show fifteen years earlier to invalidate valuable patent claims, tarnish the reputations of a senior scientist and patent attorney through a finding of inequitable conduct, and subject 3M to a $26 million *Walker Process* antitrust award consisting almost entirely of TransWeb's trebled attorney's fees. This Court should reverse this unprecedented and unjustified result.

More than sixteen months after filing its declaratory judgment action challenging 3M's patents, TransWeb made a startling new allegation: it claimed that its president had publicly distributed samples of oil-resistant, plasma-fluorinated respirator filter media (T-Melt P) at a trade show fifteen years earlier. TransWeb based this new allegation on the testimony of its president, who had a direct stake in the outcome of the litigation. No witness corroborated his story: not a single exhibitor or attendee from among the hundreds who had attended the trade show testified to TransWeb's version of events, and TransWeb did not even call the other TransWeb employee who had attended the show. TransWeb's president alone told the story of what he allegedly distributed at the trade show, and he alone attempted to connect ambiguous documents to his claim that he distributed *plasma-fluorinated* "T-Melt P"—as opposed *to non-fluorinated* "T-Melt"—at the Expo.

This uncorroborated testimony was legally insufficient to prove public use, let alone the obviousness of 3M's significantly-improved patented method for making filter media. The district court nonetheless relied on TransWeb's alleged distribution of T-Melt P at the trade show to hold 3M's claims obvious.

But the court did not stop there. It used the same murky allegations regarding the trade show to find inequitable conduct, the most serious allegation in patent law. Inequitable conduct requires proof by clear and convincing evidence that a reference was material and that an individual with a duty of candor knew about the reference, knew the reference was material, and made a deliberate decision to withhold it, such that intent to deceive is the single most reasonable inference.

TransWeb's alleged distribution of samples at a trade show fifteen years earlier comes nowhere close to meeting that demanding standard. Not only was there no hard evidence that TransWeb distributed plasma-fluorinated samples, but the limited information known to the inventor and prosecuting attorney of 3M's patents included an email pointing out that there was "no mention of oil resistant electret filter web" at the time of the show. They also knew that TransWeb had entered into a confidentiality agreement for its oil-resistant material two months *after* the trade show, a fact squarely at odds with any alleged public dissemination of the same material at the show. Moreover, the other items the district court

arguably relied on—samples shipped *to Canada* (Racal) and a respirator sold *after the filing date* (Willson)—were not even prior art, and TransWeb conceded that they could not have supported a finding of public use in the United States.

But still the district court did not stop. It next took the murky trade show allegations and its finding of inequitable conduct and used them to affirm a $26 million *Walker Process* antitrust judgment against 3M. The standard for a *Walker Process* antitrust claim is even more stringent than the standard for inequitable conduct, and requires proof of both fraud on the patent office and all the other elements of an attempted monopolization claim under Section 2 of the Sherman Act. Indeed, in its entire history, this Court has affirmed only two *Walker Process* judgments. Here, TransWeb's *Walker Process* claim failed both because of the flaws in its public use and inequitable conduct arguments and because TransWeb defined the relevant markets to artificially exclude all other filter media used as substitutes for the patented material and all respirators purchased from foreign competitors. In addition, the $26 million antitrust judgment consisted almost entirely of trebled attorney's fees, without any showing of a link between those fees and harm to competition.

The judgment is flawed at every point in its circuitous path from TransWeb's uncorroborated and self-serving allegations regarding the trade show, through the judgments of invalidity and inequitable conduct, to the unprecedented

$26 million antitrust judgment. This Court should reverse on all points and remand for consideration of 3M's unadjudicated motion for JMOL of infringement.

STATEMENT OF FACTS

A. Respirators And Filter Technologies

A respirator protects the wearer against inhaling harmful contaminants. A1096-1097. Respirators use filters made of materials designed to allow air to pass through while reducing the number of particles that pass through with it. A1097-1100. Depending on their design and intended purpose, respirators may be disposable or reusable. A1098-1099; A1130.

Respirator filters typically work in one of two ways. Some are purely mechanical, such that the filter itself simply traps or blocks the particles. A1100. Others are electrostatically charged, such that the filter material attracts particles in the air, which themselves have a positive or negative charge. A1100; A1731-1732; A2543-2544; A2555.

Electrostatically charged filters (also known as electrets) became the most common type of respirator filters by the mid-1990s. A1106. Certain factors, however, can degrade the performance of electrostatically charged filters. *Id*. These factors include the presence of oils, which can dissipate the charge in an electret filter and significantly reduce the filter's efficiency. *Id*.

Federal regulations require that certain industrial employers provide their employees with respirators certified by the National Institute of Occupational Safety and Health (NIOSH). A1105; 29 C.F.R. § 1910.134(d)(1)(ii). NIOSH's

certification standards distinguish among respirator filters based on the filter's efficiency in screening out particles (available standards include 95%, 99%, and 100% efficient), as well as the resilience of that efficiency in the presence of an oily substance. A designation of "N" indicates that the filter is not resistant and will degrade in oily conditions; "R" indicates that the filter is oil-resistant; and "P" indicates that the filter is oil-proof. A1107; 42 C.F.R. pt. 84. A P95 filter, for example, blocks approximately 95 percent of particles from penetrating the filter and will not degrade in an oily environment.

There are many filter manufacturers (*see* A2184-2185; A2189; A2230-2231; A5519; A5413-5418; A5407), and they have addressed the problem of oily environments to achieve a "P" or "R" rating in multiple ways (A2565-2567; A15094-15231). These solutions include adding extra layers of filter material, using a mechanical filter such as fiberglass, and treating the electret material so it

retains its charge in the presence of oil. A225; A1109-1110; A1126-1128; A2565-2567; A15094-15231. This case concerns one of those solutions.

B. 3M's Invention

3M's patents-in-suit disclose methods of making and using nonwoven polymeric web that is plasma-fluorinated. The asserted claims require that the plasma-fluorinated web also be hydrocharged or exceed a particular quality factor. Each of these features is discussed in turn:

Nonwoven polymeric web. Nonwoven polymeric web is a filter media made of polymers (e.g., polyethylene, polypropylene) that is commonly produced through a "meltblown" technique in which polymers are melted in an extruder, pushed out under high pressure, and collected and cooled to form a fibrous web.

Plasma Fluorination. One way of imparting oil resistance to filter media is through fluorination. A1005; A1156; A1164; A1608. 3M began experimenting with fluorination of filter media in 1982. A1606; A2502-2503; A7396. One technique involved mixing fluorinated molecules and polymer before extruding a web of fluorinated media. A1034; A1517-1522; A6861-6867. The patents-in-suit disclose another technique—"plasma fluorination"—in which nonwoven polymeric web is placed in a chamber with fluorine gas and subjected to high levels of energy that break down the fluorine molecules and deposit fragments onto the surface of the media. A1606-1608; A2097.

Hydrocharging. The other key technology in this litigation is hydrocharging, which is the process of contacting filter material with water in a manner sufficient to impart an electrical charge and then drying it. Dr. Marvin Jones, a co-inventor of the patents-in-suit, is also a co-inventor of 3M's pioneering patent on hydrocharging, which issued in March 1996. A5066-5078; *see also* A1736; A1776; A1730; A2074.

Quality Factor. The measurement of a filter's overall performance—taking into account both efficiency and breathability—is known as its "quality factor." A1780-1781; A2552. A filter's efficiency is assessed by the percentage of captured particles. A1101. A filter's breathability is measured by the difference in air pressure on opposite sides of the filter, commonly known as "pressure drop." Filters with a lower pressure drop are easier to breathe through and can generally be used longer. A1103-1104. A filter's quality factor is a function of both its efficiency in preventing penetration of particles (the greater the efficiency or the lesser the penetration, the higher the quality factor) and its breathability (the lower the pressure drop, the higher the quality factor). A1733-1735; A1781; A1759-1760; A1777.[1] Filter materials that have higher quality factors screen out a greater proportion of particles for a given level of breathability. A2553.

[1] Specifically, quality factor is the negative value of the natural logarithm of the penetration expressed as a decimal, divided by the pressure drop. A1777; A226. It is measured in the inverse of millimeters of water, or /mmH_2O. *Id.*

Unexpected Synergy. 3M was the first to discover the unexpected synergy between plasma fluorination and hydrocharging. Dr. Jones made this discovery through a series of experiments beginning in 1995. A1743-1748; A8404-8410; A2520-2522. By March 1996, Dr. Jones obtained the highest quality factor he had ever seen in oil-resistant media—above 2.0/mmH_2O—leading him to write "This is remarkable!!" in his laboratory notebook. A7539-7540; A1760; A2520-2524. Dr. Jones had "never seen anything like" it. A1760-1761.

These results were unexpected for several reasons. *First*, 3M's hydrocharging patent had suggested that hydrocharging should *not* be combined with exposure to ultraviolet light, a necessary part of the plasma fluorination process. A2109; A2062-2063; A2097; A2108-2109; A5070. *Second*, contact with water would normally be expected to diminish the performance of charged fluorinated media, not enhance it, because water would be expected to dissipate charge. *See* A1260; A2660. *Third*, the idea of "treating a waterproof material with water" was counterintuitive. A2660; *see also* A2534-2535.

3M's Patents. The patents-in-suit claim various aspects of 3M's invention. U.S. Patent No. 6,397,458 ("the '458 patent") discloses and claims methods of making and charging plasma-fluorinated polymeric web. U.S. Patent No. 6,808,551 ("the '551 patent") discloses and claims methods of filtering contaminants using plasma-fluorinated polymeric web. Both patents claim priority

to an application dated July 2, 1998 (A221; A235) and thus share a "critical date" of July 2, 1997.

The only claims that 3M asserted at trial were method claims 31 and 57 of the '458 patent. Claim 31 depends on claim 18, which states:

> 18. A method of making an electret article, which method comprises the steps of:
>
> (a) providing a nonwoven fibrous web, wherein the fibers comprise a polymeric nonconductive thermoplastic resin;
>
> (b) placing fluorine atoms on the surface of the polymeric fibers by transferring a fluorine-containing species from a gaseous phase to the fibers; and
>
> (c) charging the fluorinated nonwoven fibrous web,
>
> the charged nonwoven web comprising at least about 45 atomic % fluorine as detected by ESCA.[2]

A230-231. Claim 31 specifies that "the charging step includes hydrocharging." A231. Claim 31 thus requires (1) a nonwoven polymeric web, (2) plasma fluorination that achieves the specified level of surface fluorination, and (3) hydrocharging.

Claim 57 is an independent claim with the same elements as claim 18 except that, rather than specifying the amount of fluorine, it requires a "Quality Factor of at least about 1.0/mmH_2O." A232. Claim 57 thus requires (1) a nonwoven

[2] Electron spectroscopy for chemical analysis (ESCA) uses X-rays to measure the surface chemistry of material.

polymeric web, (2) plasma fluorination, (3) charging, and (4) a quality factor of at least about 1.0/mmH_2O.

C. TransWeb's Early Filter Media

TransWeb was founded in October 1996. A1157. Early on, TransWeb produced two main lines of products. A1194-1195; A1561; A1562; A1721-1722; A5172-5173; A2317. "T-Flo" was a non-fluorinated, split-fiber ("[f]ibrillated") material. A5172; A1274; A1721-1722; A1460. "T-Melt" referred to nonwoven meltblown media. A1384; A5173.

TransWeb eventually developed nonwoven meltblown media that was plasma-fluorinated under a confidentiality agreement with a company called Fourth State. A1162-1163; A1172. To distinguish this product from its earlier T-Flo and T-Melt products, TransWeb called it "T-Melt P" (e.g., T-Melt 30P, T-Melt 50P). A10887-10888.

The key differences among T-Flo, T-Melt, and T-Melt P for purposes of this appeal are laid out in the following chart:

	T-Flo	**T-Melt**	**T-Melt P**
Fiber Type:	Split-fiber (i.e., fibrillated) nonwoven polypropylene web. A1274; A1561-1562; A5172.	Meltblown nonwovens, such as polypropylene, polyester, or nylon web. A5173.	Meltblown nonwoven polypropylene web. Produced in different densities (e.g., T-Melt 30P, T-Melt 40P, and T-Melt 50P). A1199.
Fluorination:	Not plasma-fluorinated. A1460.	Not plasma-fluorinated, except T-Melt P products. A1384; A5173.	Plasma-fluorinated.

None of these early products was hydrocharged as required by claim 31 or achieved the quality factor required by claim 57. A1712; A9178; A1261; A1270.

D. 3M's Investigation And Disclosure

1. 3M's Investigation Regarding T-Melt P

In early 1998, 3M acquired the assets of Racal Filter Technologies, Inc. ("Racal"), a company based in Canada. A9179; A2307; A2308. Racal possessed samples of two versions of TransWeb's T-Melt P product (T-Melt 30P and T-Melt 50P). Those samples had been provided under a Confidential Disclosure Agreement (CDA) to Pierre Legare, a Racal scientist in Canada. A9178; A5115; A2333-2334.[3]

After the 1998 Racal acquisition, Legare told Dr. Jones and another colleague that TransWeb had previously given Racal samples of an oil-resistant media. A1664. Dr. Jones subsequently requested samples from Racal and had them tested at 3M. A1669. The testing demonstrated that the samples were similar to the plasma-fluorinated material on which Dr. Jones had filed a patent application several months before, except they had a much lower quality factor and did not appear to be hydrocharged. A9178; A9183-9184; A1672-1676; A1712.

[3] T-Melt 50P was simply a "thicker version" of T-Melt 30P. A2334; *see also* A1199-1200.

After seeing these results, Dr. Jones contacted Legare to learn more about the samples, and Legare told him that they were covered by a CDA. A1691-1694; A1725-1726. Dr. Jones then informed Karl Hanson, an in-house 3M patent lawyer, about the Racal samples. A1726-1727; A1932. Hanson discussed the samples with Legare and Dr. Jones, obtained copies of the CDA from Legare, and obtained copies of Legare's notes from the June 2 meeting at which the CDA was signed. A1852-1854; A1937-1943; A1954-1958; A1963-1964; A2336-2337; A2384-2385; A9178-9185.

2. Hanson's Disclosure Of T-Melt P To The PTO

On November 1, 2000, Hanson disclosed the results of 3M's investigation to the Patent and Trademark Office (PTO). He described TransWeb's T-Melt 30P and provided the PTO with a copy of the 1997 CDA, 3M's test results on the Racal sample, the quality factor derived from TransWeb's data sheet for the sample, the historical background on how 3M came into possession of the sample, and an April 28, 1999 letter from Hanson to TransWeb's president Kumar Ogale. A9178-9185. The disclosure also stated that the applicants were "unaware of any public disclosure of the Tmelt 30P product before the July 2, 1998 filing date" but "believe … the product may have been subsequently commercialized by Transweb." A9179. The examiner initially rejected 3M's claims based on the

information disclosed by Hanson and Dr. Jones, but withdrew that rejection when 3M clarified that the Racal sample had not been public. A9199; A9185.

E. Pre-Trial Proceedings

In 2010, 3M sued TransWeb for infringement in Minnesota, but dismissed the suit after TransWeb contested personal jurisdiction. *See 3M Innovative Props. Co. v. TransWeb LLC*, No. 10-cv-2132 (D. Minn.). While the Minnesota action was pending, TransWeb brought suit in New Jersey seeking a declaratory judgment that 3M's patents were invalid and unenforceable. 3M counterclaimed for infringement.

The district court denied the parties' cross-motions for summary judgment on October 15, 2012. A20338. At the same hearing, the court made clear that it was "not reading 51 claims to this jury" (A20357), informed the parties that there would be time limits on the presentation of evidence (A20390-20391), and urged the parties to reduce the number of claims for trial (A20355-20358). 3M thereafter stipulated to the dismissal of all but claims 31 and 57 of the '458 patent to streamline its trial presentation and focus on its hydrocharging and high quality factor claims. A20415-20418.

F. TransWeb's Obviousness And Inequitable Conduct Claims

TransWeb's obviousness argument depended on showing (1) public use of plasma-fluorinated T-Melt P before the critical date, and (2) the obviousness of

combining T-Melt P with hydrocharging to achieve 3M's claimed quality factor. TransWeb's inequitable conduct argument also depended on showing public use of T-Melt P, as well as that Hanson and Dr. Jones knew of that public use, knew it was material, and withheld it from the PTO with specific intent to deceive.

1. Alleged T-Melt P Samples At The 1997 Expo

TransWeb's principal witness at trial was its president, Kumar Ogale. TransWeb's invalidity, inequitable conduct, and antitrust cases all centered on Ogale's alleged distribution of plasma-fluorinated, polymeric filter media samples at the American Filtration and Separations Society's Expo ("the Expo") in Minneapolis in April 1997. Although TransWeb pled both invalidity and inequitable conduct, its complaint and two amended complaints never alleged that TransWeb had displayed or distributed plasma-fluorinated filter media at the Expo. *See* Dkts. 1, 18, 104. TransWeb's five rounds of invalidity contentions likewise made no mention of displaying or distributing plasma-fluorinated material at the Expo. A20017-20019. It was not until sixteen months after filing suit, following the close of fact discovery, that TransWeb first alleged public use at the Expo fifteen years earlier.

It is undisputed that Ogale attended the Expo, but the only witness who testified that Ogale distributed samples of *plasma-fluorinated* T-Melt P—as opposed to *non-fluorinated* T-Melt—was Ogale himself. A1382. TransWeb did

not call anyone to corroborate Ogale's story: not Dennis Durkin, the other TransWeb employee who attended the Expo (A1197); not any of the people who operated booths near TransWeb's; and not any of the more than 675 registered attendees (A6881-6927).

TransWeb also presented no photos from the Expo, did not retain a sample of what it claimed to have distributed, said it had thrown out the placard from its booth, and had no record of who had stopped by its booth. A1205-1207. Of the documents TransWeb did present, those relating to TransWeb's participation in the Expo mentioned only "T-Melt" and "T-Flo"; *none* mentioned *T-Melt P, plasma fluorination, or oil resistance*. A11048; A6860; A6876; A6879; A15008-15009; A15913; A10992. Conversely, other TransWeb documents from around the time of the Expo that referred to T-Melt P or plasma fluorination did not say anything about it being distributed *at the Expo*. A5297-5376; A10888-10890. In each instance, TransWeb relied solely on Ogale's testimony to interpret the documents or try to link them to the Expo.

TransWeb's effort to show that Hanson and Dr. Jones knew that T-Melt P had been distributed at the Expo and intentionally concealed that fact from the PTO was even more tenuous. Hanson did not attend the Expo. *See* A6881-6927. Dr. Jones attended, but he did not visit the TransWeb booth (A1720-1722; A1782), and Ogale did not testify that he had seen Dr. Jones at the Expo. The handful of

documents about the Expo available to Hanson and Dr. Jones when they were prosecuting the patents-in-suit did not mention T-Melt P, plasma fluorination, or oil resistance. A6859-6860; A11047-11048. In fact, during Hanson's investigation after learning about the Racal samples, a colleague emailed Hanson and Dr. Jones to note that while TransWeb had participated in the 1997 Expo, there was "*no mention of oil resistant electret filter web*" at the time of the Expo, meaning that it appeared TransWeb had *not* shown its oil-resistant material at the Expo. A6859 (emphasis added).

2. Racal Samples

TransWeb originally sent a CDA to Racal in December 1996, shortly after a meeting to discuss TransWeb's products. A2318; A2314; A15242-15244; A1214; A15010. The CDA was accompanied by a cover letter stating that "[o]nce [TransWeb is] in receipt of this signed document, samples will be forwarded to you for your review." A15010. On June 2, 1997, Ogale and Legare met in Canada and executed the CDA, which had been narrowed to clarify that it was intended to cover only "oily resistant thermoplastic media technology" and would have a three-year term. A15242-15244.

At trial, Ogale claimed that he had sent T-Melt P samples to Racal in Canada before the CDA was executed. But the shipping records TransWeb presented

made no reference to T-Melt P (A5154-5156), and Ogale admitted that at times he had shipped Racal non-confidential T-Flo samples (A1214).

Legare testified that he received T-Melt P samples at the June 2, 1997 meeting and did not remember receiving any T-Melt P earlier. A2354; A2377-2389. There was no evidence that Hanson or Dr. Jones knew about any T-Melt P samples sent to Racal before the confidentiality agreement, even if they existed. In addition, it was undisputed that all shipments to Racal were shipments *to Canada*. A5154-5156.

3. Willson Respirator

In November 1998, several months after Dr. Jones' patent application was filed, Dr. Jones became aware that Willson, a respirator manufacturer, was using a fluorinated media filter. A1248-1249; A1647; A6946. TransWeb conceded that it did not sell plasma-fluorinated media to Willson until September 1997, two months after the critical date. A20714-20715. There is no evidence that Hanson and Dr. Jones were aware of any sales of the Willson respirator (or the filter media used in the respirator) before the filing date. *See* A1647; A1653-1654; A1799-1800; A1846; A1860-1861.

G. TransWeb's *Walker Process* Antitrust Claim

TransWeb's antitrust claim under *Walker Process Equipment, Inc. v. Food Machinery & Chemical Corp.*, 382 U.S. 172 (1965), centered on its assertion that,

by enforcing its patents, 3M attempted to monopolize two separate markets: an "upstream" market for fluorinated polymeric material (which only TransWeb and 3M make), and a "downstream" market for NIOSH-certified P&R respirators purchased in the United States (which use many different types of filter media). TransWeb's definition of the upstream market excluded all filter media for P&R respirators other than fluorinated polymeric material, even though P&R respirator manufacturers undisputedly can and do substitute other media. A1135-1136; A1140; A2565-2567; A15094-15231. TransWeb's definition of the downstream market excluded all P&R respirators purchased from manufacturers outside the United States, even though respirators are supplied and sold worldwide.

H. JMOL And Judgment

1. 3M's Motion For JMOL And The Jury Verdict

At the close of TransWeb's case, 3M moved for JMOL on invalidity, inequitable conduct, antitrust liability and damages, infringement, and willfulness. Dkts. 492, 493, 494, 497. The district court reserved judgment. A2302.

The jury found that 3M's patent claims were invalid as obvious, that 3M committed a *Walker Process* antitrust violation, and that TransWeb did not infringe. A218-220. The jury also returned an advisory verdict finding inequitable conduct. A218. The jury ruled in 3M's favor on one of the two antitrust claims

and found that 3M had not engaged in sham litigation in pursuing its claims against TransWeb. A219.

3M renewed its JMOL motions, and the district court submitted the calculation of damages to a Special Master. Dkts. 497, 523.

2. The District Court's Decision

The district court denied 3M's motions for JMOL and held the '458 and '551 patents unenforceable due to inequitable conduct. A45.

Invalidity. The court ruled that a jury could have found that Ogale distributed samples of plasma-fluorinated material at the Expo. A9-14. The court also rejected 3M's argument that Ogale's testimony was uncorroborated. A11-14. The court, however, did not address whether it would have been obvious to hydrocharge plasma-fluorinated media to achieve the quality factor claimed by 3M, even though 3M had briefed the issue. A20424-20425.

Inequitable Conduct. The court's inequitable conduct ruling focused primarily on Hanson's and Dr. Jones's perceived failure to disclose the alleged Expo samples to the PTO. A16-34. The court also faulted Hanson and Dr. Jones with respect to the Racal sample and the Willson respirator, but acknowledged that any inequitable conduct finding premised on the Racal sample would be a "much closer call" because Hanson and Dr. Jones had an argument that it was covered by the Racal CDA. A33. The court did not address 3M's argument that a shipment to

Racal *in Canada* and a sale made by Willson *after the filing date* were not material (A20446-20447), or TransWeb's concession that Racal and Willson could not support a finding of public use (A20713-20715; A20523).

Antitrust. On the *Walker Process* claim, the court held that the jury could have properly limited the upstream product market to "fluorinated polymeric material for respirators" (A38-40) and could have properly limited the geographic market to the United States (A36). The court also held that TransWeb's trebled attorney's fees were recoverable as antitrust damages (A41) and affirmed the Special Master's calculation of those fees (A42-45).

The court entered judgment awarding TransWeb over $26 million in damages, consisting of over $22.9 million in trebled attorney's fees related to the patent claims; over $3.1 million in attorney's fees, not subject to trebling, for the prosecution of its antitrust claims; and $34,412 in lost profits (trebled to $103,236). A50-51. The court denied 3M's motion for a new trial. A52.

SUMMARY OF ARGUMENT

The district court improperly concluded that the self-serving and uncorroborated testimony of TransWeb's president regarding samples allegedly distributed at the Expo fifteen years earlier was sufficient to support a judgment of invalidity. The court then took this untenable invalidity finding and concluded that the co-inventor and prosecuting attorney of 3M's patents had known about the

purported Expo samples, had known they were invalidating prior art, and had intentionally withheld them from the PTO with intent to deceive. The court then took this untenable inequitable conduct finding and used it to support an even more extreme result, an exceedingly rare *Walker Process* antitrust judgment.

By the end, what had started as a straightforward patent case had been turned on its head based on the uncorroborated story of an accused infringer about long-past events. The district court built a house of cards on that story, with each judgment against 3M resting on an increasingly wobbly foundation. But each floor collapses under its own weight, and with it brings down the claims built upon it. This Court should reverse and remand for consideration of 3M's unadjudicated motion for JMOL of infringement. In the alternative, the Court should vacate and remand for a new trial.

Obviousness. To prove obviousness, TransWeb needed to show by clear and convincing evidence that (1) plasma-fluorinated T-Melt P—as opposed to non-fluorinated T-Melt—was in public use before July 2, 1997, and (2) it would have been obvious to combine T-Melt P with hydrocharging to achieve the quality factor claimed by 3M. TransWeb's argument fails on both points.

First, TransWeb failed to corroborate its president's allegation—made for the first time more than sixteen months into the litigation—that he had displayed and distributed samples of plasma-fluorinated T-Melt P at the Expo fifteen years

earlier. The corroboration requirement applies with "special force" to interested witnesses like Ogale. But no witness corroborated Ogale's claim, and the documents that TransWeb offered were insufficient to show that TransWeb distributed T Melt P at the Expo. The documents that referred to the Expo made no mention of T-Melt P, plasma fluorination, or even oil resistance. Instead, they mentioned only "T-Melt," which Ogale conceded could refer to *non-fluorinated* and *non-oil-resistant* products. Other documents showed what TransWeb was doing before and after the Expo, including its work on plasma fluorination, but failed to corroborate Ogale's claim that he actually distributed plasma-fluorinated material *at the Expo*. Indeed, agreements in which TransWeb asked its customers to treat plasma-fluorinated T-Melt P as confidential *after the Expo* rebut Ogale's claim that he had previously made the material public.

Second, TransWeb did not establish by clear and convincing evidence that it would have been obvious to combine plasma-fluorinated media with hydrocharging to achieve the quality factor claimed by 3M. Remarkably, however, the district court cut short its analysis after discussing public use without *any discussion* of this Court's obviousness jurisprudence or whether it would have been obvious to combine plasma-fluorinated media with hydrocharging to achieve the claimed quality factor.

Inequitable Conduct. The district court found inequitable conduct based on (1) samples allegedly distributed fifteen years earlier at the Expo that even TransWeb did not mention in its invalidity contentions; (2) samples shipped *to Canada* that were, by the court's own admission, arguably covered by a confidentiality agreement; and (3) a respirator for which there was no proof of any sales until *after the filing date*. None of these items comes close to proving by clear and convincing evidence that Hanson and Dr. Jones withheld material information, knew it was material, and acted with specific intent to deceive the PTO.

Even on a complete trial record, TransWeb failed to corroborate that it distributed T-Melt P at the Expo. The district court nonetheless stacked inference on inference to conclude that Hanson and Dr. Jones, who were unaware of most of the information presented at trial, knew there had been public use at the Expo and intended to deceive the PTO by withholding that fact. Indeed, the sheer number of instances in which the district court made an unreasonable inference or chose the least favorable inference from among multiple options shows that intent to deceive was anything but the single most reasonable inference.

The district court's other grounds for finding inequitable conduct fare no better. TransWeb conceded that neither samples shipped to Racal *in Canada* nor filter media sold to Willson *after the critical date* could have supported the jury's

verdict of invalidity. They were therefore not material and cannot support the judgment of inequitable conduct.

***Walker Process*.** Reversal of the inequitable conduct judgment requires reversal of the *Walker Process* antitrust judgment. The antitrust judgment also fails because TransWeb did not demonstrate that either of its two proposed markets is a relevant antitrust market or that trebled attorney's fees were appropriate antitrust damages.

First, TransWeb's attempt to limit the upstream product market to fluorinated polymeric media ignores the undisputed evidence that respirator manufacturers can and do substitute various different filter media to meet the NIOSH standard.

Second, TransWeb's attempt to limit the downstream geographic market to the United States ignores the undisputed evidence that respirator customers turn to suppliers outside the United States for P&R-certified respirators. Moreover, TransWeb's expert admitted that he did "no calculations" to support his opinion regarding the geographic market and did not know basic facts regarding the worldwide trade in P&R respirators.

Third, almost all of the antitrust damages in this case consisted of trebled attorney's fees. Those fees are not proper antitrust damages because TransWeb

never linked them to any harm to competition. In fact, TransWeb admitted it did not have to raise prices or exit the market due to the legal fees in this lawsuit.

STANDARD OF REVIEW

The denial of a JMOL motion is reviewed "de novo, applying the law of the regional circuit." *American Calcar, Inc. v. American Honda Motor Co.*, 651 F.3d 1318, 1341 (Fed. Cir. 2011). In the Third Circuit, courts assess "whether there is evidence upon which a reasonable jury could properly have found its verdict." *Gomez v. Allegheny Health Servs. Inc.*, 71 F.3d 1079, 1083 (3d Cir. 1995). Underlying questions of law are reviewed "under a plenary standard of review." *ZF Meritor, LLC v. Eaton Corp.*, 696 F.3d 254, 268 (3d Cir. 2012).

The district court's ultimate determination of inequitable conduct is reviewed for abuse of discretion, and underlying factual determinations are reviewed for clear error. *Star Scientific, Inc. v. R.J. Reynolds Tobacco Co.*, 537 F.3d 1357, 1365 (Fed. Cir. 2008).[4]

The denial of a motion for a new trial is reviewed for "'abuse of discretion unless the [district] court's denial of the motion is based on application of a legal precept, in which case [] review is plenary.'" *Siemens Med. Solutions USA, Inc. v. Saint-Gobain Ceramics & Plastics, Inc.*, 637 F.3d 1269, 1278 (Fed. Cir. 2011) (quoting *Curley v. Klem*, 499 F.3d 199, 206 (3d Cir. 2007)).

[4] Although this standard is compelled by circuit precedent, 3M reserves the right to argue for de novo review, if necessary, via rehearing en banc or certiorari.

A judgment of invalidity must be supported by "clear and convincing evidence." *Microsoft Corp. v. i4i Ltd. P'ship*, 131 S. Ct. 2238, 2246 (2011). A judgment of inequitable conduct requires clear and convincing evidence of materiality, knowledge of materiality, and a deliberate decision to deceive. *Therasense, Inc. v. Becton, Dickinson & Co.*, 649 F.3d 1276, 1290 (Fed. Cir. 2011) (en banc). Intent to deceive may be found only if "specific intent to deceive" is the "single most reasonable inference able to be drawn from the evidence," such that the evidence "*require[s]* a finding of deceitful intent." *Id*. at 1290-1291 (citation omitted). If more than one reasonable inference is possible, "intent to deceive cannot be found." *Id.*

ARGUMENT

I. THE INVALIDITY JUDGMENT SHOULD BE REVERSED

TransWeb's theory that claims 31 and 57 of the '458 patent were obvious depended on showing two things by clear and convincing evidence: (1) plasma-fluorinated T-Melt P was in public use under 35 U.S.C. § 102(b) before July 2, 1997, and (2) it would have been obvious to hydrocharge the material (as required by claim 31) and achieve a quality factor of at least about 1.0/mmH_2O (as required by claim 57). Not only does TransWeb's evidence fail as a matter of law on both points, but the district court did not even consider the hydrocharging and quality factor limitations in its JMOL analysis.

A. TransWeb Did Not Prove That Plasma-Fluorinated Polymeric Material Was In Public Use Before The Critical Date

The district court's invalidity opinion was based exclusively on TransWeb's alleged distribution of T-Melt P at the 1997 Expo, two months before the critical date. A9-14. In fact, TransWeb admitted below that the jury's verdict "had to be based … on the expo." A20713. TransWeb's argument, however, rested solely on Ogale's uncorroborated testimony. No evidence confirmed Ogale's claim that he distributed samples of plasma-fluorinated T-Melt P—rather than non-plasma-fluorinated T-Melt—at the Expo. This reliance on the uncorroborated testimony of an interested witness "'is insufficient as a matter of law to establish invalidity.'" *Union Carbide Chems. & Plastics Tech. Corp. v. Shell Oil Co.*, 308 F.3d 1167, 1189 (Fed. Cir. 2002) (quoting *Finnigan Corp. v. ITC*, 180 F.3d 1354, 1370 (Fed. Cir. 1999)).

"[C]orroboration of oral evidence … is the general rule in patent disputes." *Woodland Trust v. Flowertree Nursery, Inc.*, 148 F.3d 1368, 1371 (Fed. Cir. 1998). "The law has long looked with disfavor upon invalidating patents on the basis of mere testimonial evidence absent other evidence that corroborates that testimony." *Finnigan*, 180 F.3d at 1366. This Court has explained:

> The Supreme Court recognized over one hundred years ago that testimony concerning invalidating activities can be "unsatisfactory" due to "the forgetfulness of witnesses, their liability to mistakes, their proneness to recollect things as the party calling them would have them recollect them, aside from the temptation to actual perjury."

Id. (quoting *The Barbed-Wire Patent*, 143 U.S. 275, 284 (1892)).

The corroboration requirement applies regardless of whether a fact-finder could otherwise deem a witness's testimony credible. *See, e.g.*, *Juicy Whip, Inc. v. Orange Bang, Inc.*, 292 F.3d 728, 743 (Fed. Cir. 2002) (holding that testimony was uncorroborated as a matter of law but noting that "[w]e do not conclude that the witnesses below were not credible"); *Medichem, S.A. v. Rolabo, S.L.*, 437 F.3d 1157, 1170 (Fed. Cir. 2006); *id.* at 1171-1172 ("Even the most credible inventor testimony is a fortiori required to be corroborated by independent evidence.").

The corroboration requirement applies even to disinterested witnesses. *See Finnigan*, 180 F.3d at 1369; *Dow Chem. Co. v. Mee Indus., Inc.*, 341 F.3d 1370, 1378 (Fed. Cir. 2003) ("Corroboration is required of any witness whose testimony alone is asserted to invalidate a patent, regardless of his or her level of interest." (internal quotation marks omitted)). But it applies with "special force" where, as here, the witness has an interest in the outcome of the litigation and claims to have created prior art. *Finnigan*, 180 F.3d at 1368; *Medichem*, 437 F.3d at 1170; *Price v. Symsek*, 988 F.2d 1187, 1194 (Fed. Cir. 1993).

This case demonstrates the importance of strictly applying the corroboration requirement. As president of TransWeb, Ogale is a textbook example of an interested witness. He had a personal and financial stake in seeing 3M's patent claims invalidated. He was testifying about events that allegedly happened more

than fifteen years before trial. Moreover, Ogale's assertions regarding the Expo surfaced late in the litigation. Ogale did not disclose anything regarding the Expo when he prosecuted his own related patents. A8575-8599; A5377-5380. And although TransWeb amended its invalidity contentions no fewer than five times over the course of discovery, it never once indicated that Ogale had distributed samples of T-Melt P at the Expo. A20017-20019.

Despite the need for corroboration, TransWeb's allegation that it distributed samples of plasma-fluorinated T-Melt P at the Expo rested exclusively on Ogale's testimony. No other witness came forward with any testimony about what was actually distributed at the TransWeb booth. TransWeb chose not to call the other TransWeb employee who attended the Expo with Ogale. A1162; A1197. In fact, TransWeb failed to identify a single individual who could confirm Ogale's claim. *See* A1382.

The allegedly corroborative documents that TransWeb offered did not fill this void. Evidence offered to corroborate an invalidity claim must actually corroborate the material facts needed to support a finding of invalidity. *See, e.g.*, *Union Carbide*, 308 F.3d at 1171-1173, 1189 (although invention's use was undisputed, invalidity claim failed because testimony that use was *public* was not corroborated); *Allergan, Inc. v. Apotex Inc.*, 754 F.3d 952, 968 (Fed. Cir. 2014) (document showing eyelash growth produced by eyedrops did not corroborate

"claimed invention of topical application for intended therapeutic effect"); *In re Garner*, 508 F.3d 1376, 1381 (Fed. Cir. 2007) ("[C]orroboration of the existence of the device is not sufficient in this case to establish corroboration of reduction to practice. It is also necessary to corroborate that the device worked for its intended purpose."); *Juicy Whip*, 292 F.3d at 743 (purchase order did not corroborate testimony because order did not indicate that items sold matched patented invention). Thus, even where independent evidence supports some parts of a testifying witness's story, that evidence is legally insufficient where there is no corroboration as to critical details needed to prove public use.

Here, the issue is not whether Ogale attended the Expo or whether he independently developed plasma-fluorinated media. Rather, TransWeb was required to corroborate that samples of *plasma-fluorinated* polymeric web (T-Melt P) were actually displayed and distributed *at the Expo*. The documents that the district court relied on in denying JMOL do not corroborate Ogale's testimony on those critical points: the documents relating to the Expo do not say anything about *T-Melt P, plasma fluorination, or oil resistance*, and the documents relating to plasma fluorination do not show that such material was publicly distributed *at the Expo*. On the contrary, TransWeb was still requesting confidentiality for its plasma-fluorinated material *after* the Expo, which refutes any claim of public disclosure.

Promotional Materials. The district court relied on two promotional documents from shortly before the Expo: a brochure advertising TransWeb's participation in the Expo (A6879) and a magazine article announcing TransWeb's entrance into the market (A6876; A6860). Neither document mentions T-Melt P, plasma fluorination, or even oil resistance. Instead, they refer only to "T-Melt," which Ogale conceded could refer to *non-oil-resistant* products. A1384; *see also* A5173 (TransWeb brochure describing T-Melt without reference to oil resistance). The promotional documents therefore fail to corroborate Ogale's claim that he distributed *plasma-fluorinated* samples at the Expo.

3M's Internal Emails. The district court also relied on two internal 3M emails related to the Expo. A6859-6860; A11047-11048. But neither corroborates Ogale's story.

At the time of the Expo, 3M's Do-It-Yourself or "DIY" division was interested in TransWeb's T-Flo materials because it needed additional capacity for its split-fiber Filtrete™ products (furnace filters, etc.). A1563-1565; A1722. 3M's DIY division accordingly met with TransWeb representatives in early 1997. A1563-1565. 3M ultimately purchased T-Flo for several months in 1998. A1274-1275.

The first email relied on by the district court circulated shortly before the Expo. It quotes the magazine article noted above and directs two DIY division

employees, Doug Sundet and Curt Christensen, to handle all communications with TransWeb at the Expo so as "not to overwhelm" TransWeb. A6860. The email does not mention T-Melt P, plasma fluorination, or oil resistance.

The second email—on which the district court relied heavily for both invalidity and inequitable conduct—was written by Christensen to others at 3M after the Expo to summarize his interactions with TransWeb and two other companies at the Expo. A11047-11049. Again, the email does not mention T-Melt P, plasma fluorination, or oil resistance. Nor does it support the district court's conclusion that Christensen and Sundet "picked up samples of the *T-Melt* product" and gave "the price range for the *T-Melt*." A11 (citing A11048) (emphasis added). Instead, the email referred to samples of *T-Flo*, TransWeb's non-fluorinated split-fiber (fibrillated) material. A1273-1274; A1460; A1562; A1721-1722; A5172. Specifically, the email states that TransWeb had "Meltblown" (T-Melt) and "Electret (fibrillated PP)" (T-Flo) production lines. A11048; *see also* A1562. It then states: "We will be getting *Electret* [T-Flo] samples at 20 x 100 yd rolls in basis weights ranging from 25-100gsm expected by 5/9. Doug has one small 8.5 x 11 sample." A11048 (emphasis added); *see also* A1564-1565. In an error that reverberated throughout its opinion, the district court both conflated T-Flo with T-Melt and failed to distinguish between T-Melt and T-

Melt P—fundamental misunderstandings that led the court to mistakenly conclude that 3M had picked up a plasma-fluorinated T-Melt P sample at the Expo.

Patent Application and Fourth State Documents. The district court also relied on an unpublished patent application that Ogale had filed at the time of the Expo (A5297-5376) and private correspondence between TransWeb and Fourth State (A5292-5295) to show that Ogale had been working on plasma-fluorinated material several months before the Expo. A11-12. These documents are not themselves prior art. More importantly, they do not say anything about what happened *at the Expo* and therefore fail to corroborate Ogale's allegation that he distributed samples of T-Melt P to the public. *See Union Carbide*, 308 F.3d at 1171-1173, 1189 (failure to corroborate testimony that an undisputed use was actually *public*).

Racal Sample (PTX-1338). The district court also relied on PTX-1338 (A10887-10890), a sample of T-Melt 30P produced from Legare's files in Canada, to conclude that TransWeb had distributed T-Melt P at the Expo. A12-13. But Ogale testified only that PTX-1338 was "similar to what [he] was handing out at the Expo," and never actually stated that it was from the Expo. A1202. Legare, who did not attend the Expo, testified that he had received the sample in Canada after the Expo, on the day he signed the June 2, 1997 confidentiality agreement between Racal and TransWeb. A2327. Legare's testimony was corroborated by

his notes from the June 2, 1997 meeting, which indicated that he would "send data and samples to John Bowers," his colleague overseas. A15003. The next day Legare sent Bowers a letter with a label that exactly matches the label on PTX-1338:

T-Melt 30P 8 Layers x 30gm/m2	T-Melt 30P 8 Layers x 30gm/m2
A10887(PTX-1338)	A15005(DTX174)

Nonetheless, ignoring the evidence that did not fit its conclusion, the district court concluded that PTX-1338 had to have been from the Expo or a sample of the same material distributed there. This leap of logic rested primarily on the assertion that Legare could not have received PTX-1338 after the confidentiality agreement was signed because Legare had sent those samples to Dr. Jones for testing, whereas PTX-1338 was "pristine." A30. The court therefore concluded that the "sample packet was either picked up at the Expo or mailed to Legare immediately at the conclusion of the Minneapolis Expo—cut from the same material that Ogale testified that he took to the Expo." A13. But the district court's conclusion fails as a matter of simple logic.

First, Ogale testified that the samples he distributed at the Expo had at least *six* layers of material. A1200-1201. The sample in Legare's file contained only *two* layers of material. A1202. Those two layers may have been "pristine," but that says nothing about whether there were other layers or whether they were

removed and tested (or sent to John Bowers). Indeed, if no layers were removed, it would conclusively prove that PTX-1338 was *not* from the Expo. And if layers were removed and tested, then PTX-1338 would be entirely consistent with the sample Legare received after signing the confidentiality agreement and sent to Dr. Jones for testing.

Moreover, even if PTX-1338 was not from the June 2, 1997 meeting in Canada, that would not prove that it was a sample of material publicly distributed earlier at the Expo. Racal received additional samples after the confidentiality agreement was signed (A2353; A5165), and TransWeb admits that it sent multiple samples to Canada (A1212-1213).[5]

In the end, the key piece of evidence relied on by the district court to conclude that T-Melt P was publicly distributed at the Expo proves no such thing. Whatever PTX-1338 is, only Ogale's testimony links it to the Expo. PTX-1338 therefore fails to corroborate Ogale's claim that plasma-fluorinated T-Melt P was actually distributed at the Expo.

Other Samples. The district court also held that Ogale's testimony about the Expo was corroborated by various documents suggesting that TransWeb

[5] The district court criticized an attorney representing 3M for stating in correspondence with TransWeb's counsel that she was "unable to confirm when Legare obtained this sample." A29. But the attorney's residual uncertainty regarding the exact provenance of PTX-1338 in no way establishes that it was a sample from the Expo.

provided samples to potential customers around the time of the Expo. A12. But these documents suffer from the same flaws discussed above: they either refer to *T-Melt*, rather than T-Melt P; fail to corroborate what happened *at the Expo* (A5381); or both (A5193; A6870; A10992; A5153-5170).[6]

Indeed, Ogale's claim that he publicly distributed T-Melt P samples at the Expo without any restrictions cannot be reconciled with the confidentiality provisions in TransWeb's subsequent correspondence. For example, a month after the Expo, on May 28, 1997, TransWeb wrote a letter to MSA, a competitor of 3M's, that mentioned "T-Melt Plus" (rather than just "T-Melt") and stated: "Please treat these samples as confidential. We also ask that you do not chemically analyze the material or discuss the attributes of these materials with any other

[6] TransWeb did not rely on any of these purported samples as independent instances of "public use." *See* A20713-20715. Rather, TransWeb's only other contention was that "in theory" the jury might have relied on an alleged offer for sale in a letter from TransWeb to the Louis M. Gerson Company the day before the critical date. A20715. The district court declined to rely on this "theory," and for good reason. The letter did not specify a time or method of delivery (A1222-1223; A5150), the quantity to be sold (A5150), or a non-contingent price, given TransWeb's inability to plasma fluorinate on its own (A1180-1181, A5150). *See Elan Corp. v. Andrx Pharms., Inc.*, 366 F.3d 1336, 1341 (Fed. Cir. 2004) ("[A] communication that fails … to include material terms is not an 'offer' in the contract sense."). Thus, it did not invite Gerson to place an order but simply requested an opportunity to "stop [by] to discuss [the] path forward." A5150. Moreover, the letter does not disclose the underlying method itself. *See TorPharm, Inc. v. Ranbaxy Pharm., Inc.*, 336 F.3d 1322, 1327 (Fed. Cir. 2003) ("[I]f the product were sold by one other than the patentee, and the process of making remained unknown, then sale of the product would not pose a statutory bar.").

suppliers as part of our confidentiality agreement." A6928. On June 2, 1997, TransWeb signed a "Non-Disclosure Agreement" with Racal. A15242-15244. And on August 25, 1997—nearly two months after the critical date and four months after the Expo—Airflow Systems, Inc. faxed TransWeb its "signature for the Non-Disclosure Agreement on TransWeb's proprietary oil mist filter media." A15249. Far from corroborating Ogale's testimony, these post-Expo confidentiality agreements rebut Ogale's claim that he freely distributed plasma-fluorinated T-Melt P publicly at the Expo.

* * *

The corroboration requirement provides a fundamental safeguard against the inherent danger that testimony based on self-interest or imperfect recollection will be used to invalidate otherwise novel and nonobvious patent claims. No witness corroborated Ogale's self-serving testimony that he publicly distributed plasma-fluorinated T-Melt P at the Expo fifteen years earlier. And for each allegedly corroborating piece of evidence, TransWeb relied on *Ogale's testimony alone* to try to link documents that fail to mention T-Melt P, or say nothing about what happened at the Expo, to TransWeb's desired inference that plasma-fluorinated T-Melt P was distributed at the Expo.

This reliance on Ogale's testimony is legally insufficient to support a finding of public use, and this error alone requires reversal of not only the invalidity

judgment but also the inequitable conduct and *Walker Process* antitrust judgments, which also depend on Ogale's uncorroborated allegation regarding public use.

B. It Was Not Obvious To Hydrocharge Plasma-Fluorinated Material To Achieve A High Quality Factor

TransWeb's obviousness argument fails for an additional, independent reason: even if there had been public use of plasma-fluorinated material before the critical date, TransWeb did not and cannot establish that it would have been obvious to *hydrocharge* plasma-fluorinated polymeric web to achieve the high quality factor in 3M's patent claims. Remarkably, even though it was undisputed that the asserted claims were not anticipated, the district court did not even consider this critical step in the analysis, let alone properly analyze the obviousness factors in *Graham v. John Deere Co.*, 383 U.S. 1, 17-18 (1966).

TransWeb's obviousness argument was based on the combination of (1) plasma-fluorinated media with (2) 3M's earlier patent on its groundbreaking work on hydrocharging. A5066-5078; A1776; A2074. TransWeb's expert conceded that the hydrocharging patent did *not* disclose or suggest hydrocharging plasma-fluorinated material. A2108. To the contrary, it taught away from hydrocharging material that has been exposed to ultraviolet light. *See* A5070 ("The electret filter media and the polypropylene resin from which it is produced *should not be subjected to any unnecessary treatment which might increase its electrical connectivity, e.g.*, exposure to gamma rays, *ultraviolet irradiation*, pyrolysis,

oxidation, etc." (emphasis added)). As TransWeb's expert conceded, such exposure to ultraviolet light is a necessary part of the plasma fluorination process. A2097; A2108; *see also* A2659-2660.

Witnesses from both sides also agreed that, at the time of the invention, water would have been expected to *diminish* the performance of charged fluorinated media, not enhance it. Ogale testified: "Typical electrets or normal electrets usually after washing they loose [sic] their performance, and so they can't be reused. Their efficiency goes down, they're not as good as they were before." A1260; *see also* A1262 (similar); A7257 (similar). Indeed, Ogale had known about 3M's hydrocharging patent since 1996 (A1417), but it nonetheless did not occur to him to experiment with wet-charging plasma-fluorinated material until November 1999 (A1260-1261; *see also* A2659). 3M's expert Dr. Glew reinforced the point that water would have been expected to dissipate charge, rather than enhance it. A2660 ("[T]hese are charged materials. Water is generally known to dissipate charge."). He further explained that it would have been counterintuitive to "treat[] a waterproof material with water." A2660.

The validity of asserted claims 31 and 57 is further confirmed by objective indicia of non-obviousness, which this Court has stressed "'may often be the most probative and cogent evidence in the record,'" *In re Cyclobenzaprine Hydrochloride Extended-Release Capsule Litig.*, 676 F.3d 1063, 1075 (Fed. Cir.

2012), and are "crucial in avoiding the trap of hindsight," *Leo Pharm. Prods., Ltd. v. Rea*, 726 F.3d 1346, 1358 (Fed. Cir. 2013). TransWeb's expert admitted that the products embodying the asserted claims had been commercially successful. A2105. The evidence of unexpected results was also overwhelming; Dr. Jones's lab notebook entry for his discovery of the synergy between hydrocharging and fluorinated media noted that this phenomenon was "remarkable!!" A7540; A1760-1761. And, as discussed above, the success 3M achieved was quite unexpected.

The district court simply ignored all this evidence of non-obviousness, which was error in itself. When the issue is actually considered, it is clear that claims 31 and 57 were not obvious at the time of invention.

II. THE INEQUITABLE CONDUCT JUDGMENT SHOULD BE REVERSED

The district court improperly found inequitable conduct based on (1) the samples allegedly distributed fifteen years earlier at the Expo; (2) samples sent *to Canada* that even TransWeb admitted could not support a finding of public use in the United States and that were sent to a company that signed a confidentiality agreement; and (3) a respirator for which there was no proof of any sales until *after the filing date*, which contained filter media that TransWeb also admitted could not support a finding of public use.

"[I]nequitable conduct is the 'atomic bomb' of patent law." *Therasense*, 649 F.3d at 1288. To prevail on such a claim, "the accused infringer must prove by

clear and convincing evidence that the applicant knew of the reference, knew that it was material, and made a deliberate decision to withhold it." *Id*. at 1290. "A finding that the misrepresentation or omission amounts to gross negligence or negligence under a 'should have known' standard does not satisfy this intent requirement." Rather, "specific intent to deceive the PTO" "must be the '*single most reasonable inference*' able to be drawn from the evidence." *Therasense*, 649 F.3d at 1290 (emphasis added). In other words, "the evidence 'must be sufficient to *require* a finding of deceitful intent in the light of all the circumstances.'" *Id*. "[W]hen there are multiple reasonable inferences that may be drawn, intent to deceive cannot be found." *Id*. at 1290-1291.

A finding of inequitable conduct must be based on the conduct, knowledge, and intent of the specific individuals with a duty of candor. *Exergen Corp. v. Wal-Mart Stores, Inc.*, 575 F.3d 1312, 1329 (Fed. Cir. 2009). It cannot be based on the collective knowledge of a corporation or group. *Id*.

A. Expo Samples

The district court abused its discretion when it concluded that Hanson and Dr. Jones committed inequitable conduct by failing to disclose TransWeb's alleged distribution of plasma-fluorinated samples at the Expo.

Materiality. TransWeb's failure to corroborate Ogale's assertion that he distributed plasma-fluorinated samples at the Expo (*supra* Section I.A) precludes a

finding of inequitable conduct, because without corroborated, clear and convincing evidence that T-Melt P was actually distributed, TransWeb cannot prove that there was any material omission regarding the Expo.

Knowledge, Knowledge of Materiality, and Intent to Deceive. Hanson and Dr. Jones could not have known about alleged Expo samples that never existed. Even if those samples had existed, Hanson and Dr. Jones knew only a tiny fraction of the Expo story on which TransWeb based its invalidity case. Faced with this fact, the district court stacked assumption on top of assumption to arrive at its inequitable conduct determination. Specifically, drawing every inference against Hanson and Dr. Jones, the district court assumed:

- TransWeb in fact distributed plasma-fluorinated samples at the 1997 Expo, even though:
    - TransWeb did not allege public use at the Expo in five rounds of invalidity contentions (A20017-20019), did not disclose the Expo when prosecuting its own patent (A8575-8878; A5377-5380), did not call any witness to corroborate Ogale's testimony (*supra* pp. 16-17, 31), and did not present any document demonstrating that the samples TransWeb distributed at the Expo were T-Melt P (*supra* pp. 17, 31-40).
- Dr. Jones had actual knowledge of these alleged samples at the time (*see* A19), even though:
    - Ogale did not testify that he met Dr. Jones at the Expo, Dr. Jones testified that he did not visit the TransWeb booth (A1720-1722; A1782), and Hanson never even visited the Expo (*see* A6881-6927).
- Dr. Jones had seen and remembered an email sent shortly after the Expo by 3M employees in a different business unit, the DIY division,

and believed that it referred to plasma-fluorinated polymeric web (*see* A21-22), even though:

- the email made no reference to T-Melt P, plasma fluorination, or oil resistance (A11048), and the "Electret (fibrillated PP)" sample it mentioned was T-Flo, not T-Melt P (*supra* p. 34-35).

- With actual knowledge of the alleged Expo samples, Dr. Jones nonetheless inexplicably decided to wait until the alleged Expo samples became prior art and then deceive the PTO (A19), even though:
  - if alleged Expo samples had actually existed and Dr. Jones had actually known about them, he simply could have filed a patent application within the one-year grace period.

- After Hanson and Dr. Jones learned about the Racal sample and the Willson respirator, they believed that the same material had been distributed at the Expo (A24-25), even though:
  - their colleague's email looking back at the Expo said: "Notice that at this time there was *no mention of oil resistant electret filter web*." A6859 (emphasis added).

- Hanson and Dr. Jones knew about PTX-1338 and believed it was a sample from the Expo, even though:
  - PTX-1338 was found in Legare's files during this litigation (A2327) and there was no evidence that Hanson or Dr. Jones knew about any Racal samples other than the ones Legare said he received when he signed the TransWeb confidentiality agreement (*infra* pp. 48-49).

- Hanson and Dr. Jones made a conscious decision not to disclose the alleged Expo samples with specific intent to deceive the PTO, even though:
  - Dr. Jones is an accomplished scientist and inventor with more than 30 patents (A1508; A1715-1717), Hanson has prosecuted hundreds of patent applications in his 22-year career at 3M (A1904), and neither stood to gain or lose directly from the success or failure of the application (A1717-1718; A1913).

Each of these assumptions fails on its own. Taken together, they confirm that TransWeb's inequitable conduct claim was premised on conjecture, speculation, and inferences that are far from reasonable. Indeed, the sheer number of steps in the court's analysis and number of times that it drew contested inferences against Hanson and Dr. Jones alone confirm that intent to deceive was not "the single most reasonable" inference. It was hindsight, and faulty hindsight at that, to assume that Hanson and Dr. Jones knew of the alleged Expo samples, understood their materiality, and withheld that information with specific intent to deceive the PTO.

B. Racal Samples

The district court extensively discussed the Racal samples, but it is unclear to what extent the court relied on those samples in reaching its judgment. In fact, the court stated that "if [the Racal samples] were the only prior art, then inequitable conduct would be a much closer call." A33. To the extent the inequitable conduct judgment is based on the Racal samples, it was an abuse of discretion.

Materiality. TransWeb's attempt to establish the materiality of the Racal samples suffered from two fundamental flaws. *First*, Racal was located in Canada. Shipment of samples to Racal therefore did not establish public use *in the United States*.[7] Indeed, TransWeb conceded that the jury's verdict of invalidity could not

[7] Mere shipment of samples does not by itself establish public use. *See Minnesota Mining & Mfg. Co. v. Chemque, Inc.*, 303 F.3d 1294, 1307 (Fed. Cir. 2002). And, by definition, shipment of samples *to Canada* does not constitute

have been based on the Racal samples. *See* A20713; A20523 ("TransWeb did not argue that the samples sent to Pierre Legare constituted an invalidating public use in the United States under 35 U.S.C. § 102(a)-(b)."). The district court, however, did not even consider 3M's arguments and TransWeb's concessions on this dispositive issue, which preclude a finding of inequitable conduct.

Second, samples provided to Racal under the June 2, 1997 confidentiality agreement were undisputedly not material. TransWeb therefore had to prove that it sent samples of T-Melt P that were not covered by the agreement. The shipping records that TransWeb presented, however, failed to corroborate Ogale's assertion that the samples he sent to Racal before signing the confidentiality agreement were T-Melt P, rather than T-Flo or other non-fluorinated materials that Ogale was publicly distributing at the time. A5154-5156.[8] TransWeb thus failed to prove materiality by properly corroborated, clear and convincing evidence.

"public use … *in this country*." 35 U.S.C. § 102(b) (emphasis added). For example, *Johns Hopkins University v. CellPro, Inc.*, 152 F.3d 1342, 1366 (Fed. Cir. 1998), held that "neither export from the United States nor use in a foreign country of a product covered by a United States patent constitutes infringement." If shipping samples abroad does not qualify as *use* for purposes of infringement, it certainly does not qualify as public use for purposes of invalidity.

[8] The T-Melt 50P sample was not material for the same reasons and because it was cumulative of the Racal T-Melt 30P sample that was disclosed to the PTO. A1199-1200; A2333-2334; *see also Honeywell Int'l Inc. v. Universal Avionics Sys. Corp.*, 488 F.3d 982, 1000 (Fed. Cir. 2007) ("[i]nformation cumulative of other information already before the Patent Office is not material").

Knowledge, Knowledge of Materiality, and Intent to Deceive. Even if T-Melt P samples had been sent to Racal before the confidentiality agreement, there was no evidence that Hanson or Dr. Jones knew anything about them. The only evidence of earlier shipments of *any* kind—including earlier shipments of T-Flo—came from Ogale's testimony and TransWeb's shipping records (which do not refer to T-Melt P, plasma fluorination, or oil resistance). That testimony and those records were not available to Hanson or Dr. Jones. In addition, 3M did not even own Racal at the time, so Hanson and Dr. Jones had no direct knowledge of what had happened there. Legare did not remember receiving T-Melt P samples before the confidentiality agreement. A2349. And there was certainly no evidence that Hanson and Dr. Jones had been informed of such samples. To the contrary, Legare told Dr. Jones that the Racal samples had been provided under a confidentiality agreement. A1694.

Even if Hanson and Dr. Jones had known about any earlier samples, the district court conceded that they had an "argument that … samples sent to Legare [at Racal] without a confidentiality agreement were 'covered' by the agreement signed a few weeks later." A33. Although the court labeled that argument "weak," its existence is sufficient, by itself, to show that there were "multiple reasonable inferences" available, such that "intent to deceive" is not the "single

most reasonable inference." *Therasense*, 649 F.3d at 1290-1291.[9]

C. Willson P-95 Respirator

To the extent the district court also relied on the Willson P-95 respirator to support a finding of inequitable conduct (A33), it was an abuse of discretion.

Materiality. TransWeb conceded that the Willson respirator could not have supported a verdict of public use. *See* A20714-20715. TransWeb admitted that it did not sell any plasma-fluorinated material to Willson until after the critical date (A1249; A6940), and there was no evidence of any actual sales of the Willson respirator until after the filing date (*see* A1647). Accordingly, nothing related to the Willson respirator was "but for" material.

Knowledge, Knowledge of Materiality, and Intent to Deceive. Dr. Jones and Hanson did not learn about Willson's commercial product until November 1998. A1647-1652. They then disclosed to the PTO that, although they were not aware of any public disclosure of TransWeb's product before the filing date, "the

[9] In negotiating a new confidentiality agreement with 3M in 1999 relating to other samples, Ogale did not object to a letter from Hanson laying out 3M's understanding that samples in Racal's possession were sent as "part of" the "agreement dated June 2nd, 1997." A6952. The district court viewed this letter as a pretext and accused Hanson of failing to tell the PTO "the actual subject of the letter." A27. But Hanson's supplemental information disclosure statement never quoted the letter or otherwise presented it as an admission by Ogale, even though that was a reasonable understanding. A9178-9179. Instead, Hanson simply attached the letter that he sent Ogale as part of the procedural background. A9185. The court's inference that Hanson somehow misled the PTO with "selective quotation" (A27) from the letter that he submitted in full and never quoted from is inexplicable.

product may have been subsequently commercialized by Transweb." A9179. The district court's conclusion that "they knew it had been earlier commercialized" (A33 n.26) is completely unsupported. Even if the respirator took several months to get to market (A23 n.18), there is no evidence that Hanson and Dr. Jones knew the filter media was on the market before the filing date. Once again, intent to deceive was not the single most reasonable inference.

III. THE ANTITRUST JUDGMENT SHOULD BE REVERSED

Reversal of the inequitable conduct judgment requires reversal of the *Walker Process* antitrust judgment. The *Walker Process* judgment also independently fails because TransWeb did not present substantial evidence to support the other elements of an attempted monopolization claim. *See Walker Process*, 382 U.S. at 174 ("[E]nforcement of a patent procured by fraud on the Patent Office may be violative of § 2 of the Sherman Act provided the other elements necessary to a § 2 case are present."). In particular, TransWeb had to define proper geographic and product markets and prove that 3M "had specific intent to monopolize the relevant market" and "possessed sufficient market power to come dangerously close to success." *Barr Labs., Inc. v. Abbott Labs.*, 978 F.2d 98, 112 (3d Cir. 1992).[10] TransWeb also had to prove that it suffered antitrust injury and damages flowing

[10] Federal Circuit law applies to the existence of *Walker Process* fraud, but regional circuit law applies to the elements of the antitrust claim not unique to patent law. *See, e.g.*, *Unitherm Food Sys., Inc. v. Swift-Eckrich, Inc.*, 375 F.3d 1341, 1349 (Fed. Cir. 2004).

from the alleged harm to competition. *Mathews v. Lancaster Gen. Hosp.*, 87 F.3d 624, 641 (3d Cir. 1996) (citing *Brunswick Corp. v. Pueblo Bowl-O-Mat, Inc.*, 429 U.S. 477, 489 (1977)).

In its entire history, this Court has affirmed only two *Walker Process* judgments. This case does not rise to that extraordinary level. In fact, TransWeb's *Walker Process* theory suffers from multiple defects.

A. Without A Showing Of Inequitable Conduct, The Antitrust Verdict Cannot Stand

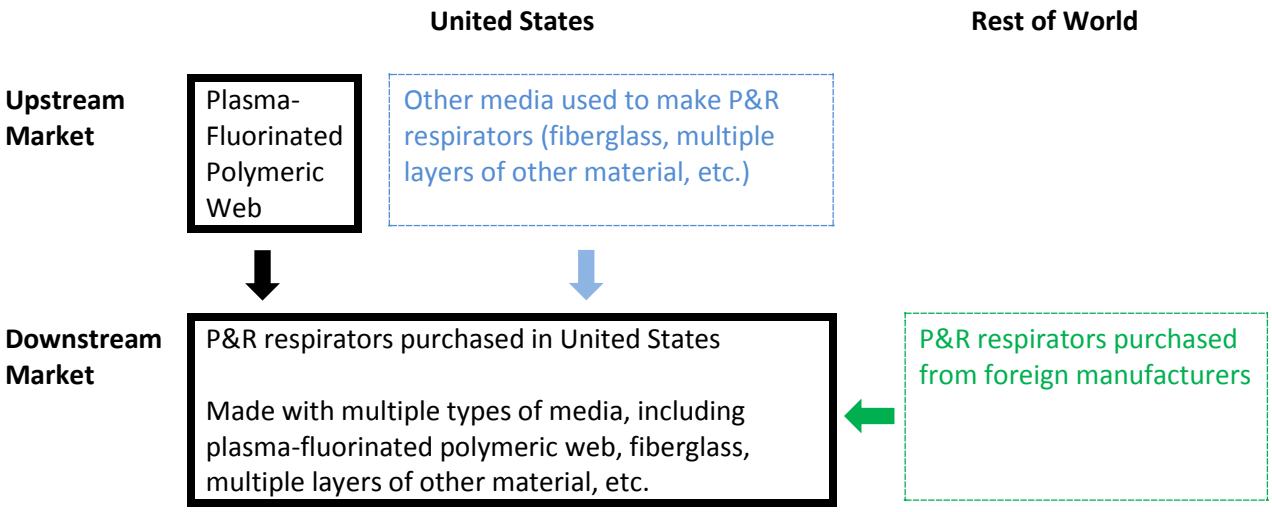
Before *Therasense*, a *Walker Process* claim required "higher threshold showings of both materiality and intent than are required to show inequitable conduct." *Dippin' Dots, Inc. v. Mosey*, 476 F.3d 1337, 1346 (Fed. Cir. 2007). Although *Therasense* brought the standards closer together, a *Walker Process* claim is at least as difficult to prove as inequitable conduct. TransWeb's failure to prove inequitable conduct therefore requires reversal of the antitrust judgment.

B. TransWeb Failed To Prove That Either Of Its Two Proposed Markets Is A Relevant Antitrust Market

The antitrust judgment also must be reversed because there is no support for TransWeb's upstream product market or its downstream geographic market. The scope of the relevant market is critical in a *Walker Process* case: "Without a definition of [the relevant] market there is no way to measure [the defendant's] ability to lessen or destroy competition." *Walker Process*, 382 U.S. at 177.

TransWeb bears the burden of proving the relevant product and geographic market. *Tunis Bros. v. Ford Motor Co.*, 952 F.2d 715, 726 (3d Cir. 1991).

At trial, TransWeb argued that 3M had attempted to monopolize two narrowly-defined markets: (1) an "upstream" market for fluorinated polymeric media, and (2) a "downstream" market for NIOSH-certified P&R respirators purchased in the United States.



But TransWeb's definition of the upstream market artificially excluded all other filter media used to make P&R respirators (blue), and its definition of the downstream market artificially excluded the numerous foreign competitors that make P&R respirators (green).

1. TransWeb's Own Evidence Disproves Any Distinct Product Market For Fluorinated Polymeric Media

A relevant product market consists of "'commodities reasonably interchangeable by consumers.'" *Tunis Bros.*, 952 F.2d at 722 (quoting *United*

States v. E.I. du Pont de Nemours & Co., 351 U.S. 377, 395 (1956)). Here, the evidence focused on the use of fluorinated polymeric filter media to manufacture P&R respirators. Thus, the question is what types of media P&R respirator manufacturers could turn to in response to a price increase for fluorinated polymeric filter media. The upstream market can only be narrowly limited to fluorinated polymeric media (as TransWeb sought) if the evidence supports a finding that customer preferences are so strong that respirator manufacturers will not substitute other media that meet the P&R standards. *See IGT v. Alliance Gaming Corp.*, 702 F.3d 1338, 1347 (Fed. Cir. 2012) (player preference for particular type of gaming machine showed only that games competed in a "differentiated market," not that the preferred game was a separate market). No such evidence was offered here.

TransWeb's own experts established that there are numerous competitors for fluorinated polymeric filter media in the upstream market. TransWeb's industrial hygiene expert admitted that P&R respirators are made with a number of different filter media. *See, e.g.*, A1135-1136 ("Q. … There are ways to achieve a P 100 respirator other than fluorine. Right? A. Yes. Q. One of them is fiberglass. Right? A. Yes. Q. One of them is pleated material, folded material. Right? A. I assume it's having more electret material, if that's done through pleating, that's

fine."); *see also* A1136 ("there are respirators on the market that are P100 that are not fluorinated"); A1140.

In addition, 3M's expert tested 75 respirators on the market that meet the P or R standard and testified without contradiction that *over 80% did not use plasma-fluorinated polymeric media*. A2565-2567; A15094-15231 (citing examples of P&R respirators with unfluorinated media from Aearo, Dräger Safety, Kimberly-Clark, Louis Gerson & Co., McCordick Glove & Safety, Mine Safety Appliances, Moldex-Metric, North Safety, Sperian Respiratory Protections, etc.).

Thus, the evidence showed that respirator manufacturers substitute all manner of filter media in the upstream market to make products that compete in the alleged downstream market for P&R respirators. A2230-2231 (TransWeb's antitrust expert admitted "there are many different types of respirators that we've heard about within [the downstream market]" and customers "could substitute within the set of P&R respirators").

Given that evidence, it is impossible to conclude that fluorinated polymeric media have no substitutes in the upstream market. *See, e.g., DSM Desotech Inc. v. 3D Sys. Corp.*, 749 F.3d 1332, 1345-1346 (Fed. Cir. 2014) (competition in the market for 3D printers precludes finding that resins for specific machines are in distinct product markets); *Unitherm Food Sys., Inc. v. Swift-Eckrich, Inc.*, 375 F.3d 1341, 1364 (Fed. Cir. 2004) (expert testimony was insufficient to support antitrust

verdict where expert concluded that patented products competed with unpatented products "at the retail level, but somehow not at the wholesale level" and "never explained this seeming anomaly").[11]

The district court's analysis of fluorinated polymeric media's perceived advantages (A38) does not establish the absence of substitutes. Courts have repeatedly rejected market definitions that rely on quality advantages. *See, e.g.*, *Brown Shoe Co. v. United States*, 370 U.S. 294, 326 (1962) (low- and medium-"price/quality" shoes in the same market); *Unitherm*, 375 F.3d at 1364 (reasonable jury could not rely on expert testimony that "described the market in terms of technological substitutability, not economic substitutability"); *Golan v. Pingel Enter., Inc.*, 310 F.3d 1360, 1369 (Fed. Cir. 2002) (rejecting distinction between markets for high-end and low-end motorcycle fuel valves given evidence of substitution). Courts have also rejected market definitions that merely track patent claims. *Delano Farms Co. v. California Table Grape Comm'n*, 655 F.3d 1337, 1352 (Fed. Cir. 2011).

Here, TransWeb offered no evidence comparing the relative performance of 3M's respirators to other respirators or mapping that performance onto their relative prices to determine "whether potential customers of the patented process

[11] The reversal of this Court's decision on procedural grounds in *Unitherm Food Sys., Inc. v. Swift-Eckrich, Inc.*, 546 U.S. 394 (2006), does not undermine its substantive analysis of the relevant product market.

faced with a price increase would shift to other processes offering different combinations of benefits." *Unitherm*, 375 F.3d at 1364. Nor could the physical differences between various types of filter media support TransWeb's market definition given that undisputed evidence showed that customers do in fact use "many different types" of filters when manufacturing P&R respirators.

2. TransWeb's Attempt To Limit The Downstream Geographic Market To The United States Fails

The relevant geographic market is the area "in which a potential buyer may rationally look for the goods or services he seeks." *Gordon v. Lewistown Hosp.*, 423 F.3d 184, 212 (3d Cir. 2005). Here, none of the evidence supported a finding that the market was limited to the United States. Instead, the uncontroverted evidence clearly showed that customers can and do turn to suppliers located outside the United States. *See, e.g.*, A2184-2185 (identifying German, Scandinavian, and Asian manufacturers that sell P&R respirators in the United States).

The district court incorrectly interpreted three pieces of evidence as supporting TransWeb's purported downstream geographic market excluding all foreign suppliers. *First*, the court conflated a U.S. safety standard with the location of manufacturers to whom customers could turn for compliant products. The court stated that "NIOSH is a U.S. agency that sets standards for respirators used by companies operating within the U.S." A36. But that is irrelevant. Nothing about

the NIOSH standards precludes foreign manufacturers from making and selling respirators that meet those standards, and such sales routinely occur (A2184-2185; A15094-15231).

Second, the court relied on several 3M documents that mentioned a "North American Market" or examined country-by-country sales. A36. Again, these documents are insufficient to support limiting the geographic market to the United States. Not only do they relate to a market broader than the United States (i.e., North America) (e.g., A5413) and sales around the world (e.g., A5516), but they focus on where 3M sells its products rather than where customers can find alternative supply sources. *See Tunis Bros.*, 952 F.2d at 726 ("[T]he geographic market is not comprised of the region in which the seller attempts to sell its product, but … the area where [its] customers would look to buy such a product."). Because the record is clear that U.S.-based customers purchase from manufacturers in Europe and Asia (e.g., A2184-2185), the record does not support a market restricted to the United States.

Third, the court relied on the testimony of TransWeb's economic expert, Dr. Reiff. A36. However, that testimony is baseless and should have been excluded. Dr. Reiff's expert report simply *assumed* that the geographic market was limited to the United States, and he never disclosed before trial that he intended to present an expert opinion regarding the proper geographic scope of the market. A2238.

Even if his testimony is considered, Dr. Reiff's bare assertion that "the U.S. was a geographic market" (A2243) is insufficient as a matter of law. Dr. Reiff admitted on cross-examination that he did "no calculations" to support his opinion and did not know basic facts regarding the worldwide trade in P&R respirators:

> Q. Dr. Reiff, I'd like to ask you just a couple of questions about what you said about geographic market earlier today. Did you do any calculations to determine that the relevant market was the United States?
>
> A. No, I did no calculations to determine that.
>
> * * *
>
> Q. Do you know what percentage of P and R respirators sold in the United States that come from outside the United States?
>
> A. I don't know the percentage.
>
> Q. Do you know the percentage of respirators, P and R respirators, made inside the United States that are shipped outside the United States?
>
> A. I don't know that.

A2299. Dr. Reiff's conclusory opinion cannot support the verdict. *See, e.g.*, *Brooke Grp. Ltd. v. Brown & Williamson Tobacco Corp.*, 509 U.S. 209, 242 (1993) ("When an expert opinion is not supported by sufficient facts to validate it in the eyes of the law, or when indisputable record facts contradict or otherwise render the opinion unreasonable, it cannot support a jury's verdict.").[12]

[12] Dr. Reiff further exaggerated 3M's alleged market power by calculating 3M's downstream market share based on a document that (1) combined data for

C. TransWeb Was Not Entitled To Trebled Attorney's Fees As Antitrust Damages

Over $22.9 million of the antitrust damages awarded in this case were TransWeb's attorney's fees, trebled. Even if TransWeb had proven its antitrust claim, such fees could not properly establish antitrust injury and be recovered as antitrust damages because TransWeb never proved any link between those fees and an impact on competition.

Neither the Third Circuit nor this Court has ever approved an award of treble attorney's fees as antitrust damages in a *Walker Process* suit. Indeed, even the pre-trial order permitting TransWeb to pursue such a theory admitted that "[w]hether litigation costs can qualify as antitrust injury is a 'controversial' proposition." A282 (quoting *Abbott Labs. v. Teva Pharms. USA, Inc.*, 432 F. Supp. 2d 408, 431 n.19 (D. Del. 2006)).

The general rule is that "'an antitrust plaintiff must prove that challenged conduct affected the prices, quantity or quality of goods or services,' not just his own welfare." *Mathews*, 87 F.3d at 641. Courts have thus held that litigation fees cannot be recovered as antitrust damages in the *Walker Process* context absent a

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disposable respirators and irrelevant non-oil-resistant "N" respirators, and (2) excluded all reusable respirators. A2284-2289. Those reusable respirators that Dr. Reiff excluded from his analysis constituted the largest segment of the market, and 3M's market share for those respirators was only 30-33%. A2194-2195. The district court, however, inexplicably got the witnesses backwards when it said that Dr. Reiff "included both disposable and reusable respirators" and 3M's witness included irrelevant N respirators. A39 & n.32.

showing that those fees affected competition. *See, e.g., Brotech Corp. v. White Eagle Int'l Techs. Grp., Inc.*, 2004 WL 1427136, at *6-7 (E.D. Pa. June 21, 2004) (litigation costs not an antitrust injury absent showing that they "had any effect on competition, on the price, quantity or quality of … products, or prevented … entry into the market"); *Varian Semiconductor Equip. Assocs., Inc. v. Advanced Ion Beam Tech, Inc.*, 2009 WL 2425849, at *5-7 (D. Mass. Aug. 4, 2009).

TransWeb presented no evidence linking its attorney's fees to any effect on competition. To the contrary, Ogale testified that TransWeb had not changed its prices due to the lawsuit (A1459), TransWeb's economic expert conceded that TransWeb had not exited the market (A2268), and the same expert admitted that he had "no opinion" on whether TransWeb's payment of legal fees had harmed competition. A2300-2301.

In addition, cases that have treated attorney's fees as antitrust damages have involved sham litigation. *See, e.g., Handgards, Inc. v. Ethicon, Inc.*, 601 F.2d 986 (9th Cir. 1979). Sham litigation is based on the premise that the patent-owner is seeking to use the litigation itself "'as opposed to the *outcome* of that process—as an anticompetitive weapon.'" *Professional Real Estate Investors, Inc. v. Columbia Pictures Indus., Inc.*, 508 U.S. 49, 60-61 (1993) (*PRE*). Here, however, the jury specifically found that 3M's litigation was not a "sham." A219. Even though 3M's infringement counterclaim was ultimately unsuccessful, the jury's finding

that it was not a sham meant that 3M had an objective basis for believing it could enforce its patent. In such circumstances, it would be particularly inappropriate to award attorney's fees as antitrust damages, because the threat of such an award would discourage patentees from raising legitimate arguments for patent validity whenever *Walker Process* claims are raised. Indeed, the Supreme Court has repeatedly held that the right to petition the courts with good-faith arguments is constitutionally protected. *See PRE*, 508 U.S. at 61 n.6 (declining to decide to what extent *Noerr* immunity applies to *Walker Process* claims).[13]

This Court should not expand the scope of antitrust liability by treating litigation costs as antitrust damages where, as here, there is no showing that those costs adversely affected competition in the relevant market.

IV. THE COURT SHOULD REMAND FOR CONSIDERATION OF 3M'S UNADJUDICATED MOTION FOR JMOL OF INFRINGEMENT

The only disputed issue regarding infringement was whether TransWeb's manufacturing process includes hydrocharging. Before this litigation, TransWeb had referred to its "rinsing" lines as "charging lines" (A1331); admitted that it uses a "wet charging process" prior to being dried (A1332-1333; *see also* A15887); and admitted that it "used water to charge the web" (A1409-1410; *see also* A1262;

[13] Although *Nobelpharma AB v. Implant Innovations, Inc.*, 141 F.3d 1059, 1071 (Fed. Cir. 1998), held that a patentee engaged in *Walker Process* fraud could be held liable for the anticompetitive effects of its suit without proof of objective baselessness, *Nobelpharma* did not hold that the accused infringer's costs of defending the suit are recoverable as antitrust damages.

A2592-2593). Unrebutted expert testimony showed that TransWeb's rinsing lines imparted a charge. A2676-2677; A2682; A2602; *see also* A2520; A1781.

The district court did not consider any of these points because it denied 3M's motion for JMOL of infringement as moot in light of its invalidity and inequitable conduct rulings. A46. In addition to reversing those rulings, this Court should remand with instructions to address 3M's motion for JMOL of infringement. Alternatively, this Court should order a new trial on all issues because, for the reasons stated herein, the judgment was against the great weight of the evidence and would result in a manifest injustice if enforced against 3M. *See Sheridan v. E.I. DuPont de Nemours & Co.*, 100 F.3d 1061, 1076 (3d Cir. 1996) (en banc).

CONCLUSION

The Court should reverse the invalidity, inequitable conduct, and antitrust judgments and remand for consideration of 3M's unadjudicated motion for JMOL of infringement, or alternatively grant a new trial on all issues.

Respectfully submitted,

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ADDENDUM

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FOR PUBLICATION

UNITED STATES DISTRICT COURT
DISTRICT OF NEW JERSEY

TRANSWEB, LLC, Plaintiff, v. 3M INNOVATIVE PROPERTIES COMPANY and 3M COMPANY, Defendants.	Hon. Faith S. Hochberg, U.S.D.J. Civil Case No. 10-4413 (FSH) (JBC) **OPINION** Date: April 21, 2014

HOCHBERG, District Judge:

I. BACKGROUND

Just breathing in a dirty or contaminated area can cause serious injury. Industrial respirators are designed to protect workers in these environments. This dispute revolves around the filtration membrane used in industrial respirators. OSHA (Occupational Safety and Health Administration) regulations govern the type and quality of these respirators, which are rated and approved by NIOSH (the National Institute for Occupational Safety and Health, a division of the Centers for Disease Control).

Both 3M and a small, new company called TransWeb LLC ("TransWeb") independently developed filtration web for use in these NIOSH-approved respirators. At issue in this case is whether 3M's patents on the filtration media are valid and infringed by TransWeb, and whether

3M violated the antitrust laws. 3M sought solely to enjoin TransWeb, and TransWeb sought damages (primarily in the amount of its attorneys' fees) and to invalidate the 3M patents.

A. Procedural History

TransWeb filed this action on August 27, 2010, against 3M Company and 3M Innovating Properties Company (collectively, "3M"), seeking a declaratory judgment of invalidity as to claims in the family of 3M patents, unenforceability of those patents due to inequitable conduct in the prosecution of those patents, and non-infringement by TransWeb. 3M counterclaimed for patent infringement.[1] 3M is the assignee of the two patents-in-suit: U.S. Patent Nos. 6,397,458 ("the '458 patent") and 6,808,551 ("the '551 patent"). The inventors on those patents are 3M employees, and a 3M in-house lawyer prosecuted the patents.

TransWeb filed an amended complaint stating antitrust claims of *Walker Process* fraud and sham litigation. (It had also claimed that 3M infringed a TransWeb patent, but TransWeb voluntarily dismissed its claims of infringement of its patent, and 3M voluntarily dismissed its counterclaims pertaining to those patents.).

Two days before the jury trial, 3M voluntarily dismissed fifty-six of its fifty-eight patent infringement claims of the '458 patent and all patent infringement claims of the '551 patent. Only two of 3M's claims in a single patent—the '458 patent—were litigated to a jury, together with TransWeb's antitrust and patent claims. 3M contended that TransWeb willfully infringed those claims.

[1] 3M had originally sued TransWeb in the District of Minnesota, but voluntarily dismissed without prejudice after a hearing was conducted regarding whether TransWeb was subject to personal jurisdiction. TransWeb then filed this action.

B. The Product and Method

At issue in this case is the method for making filtration media—specifically, plasma fluorinated, nonwoven meltblown polypropylene electret web. That material can be used in numerous filtration applications, but for the purposes of this case, the most important use of a fluorinated electret was for respirators that workers must wear in oily environments.

In 1995, NIOSH began developing new regulations for oil-proof and oil-resistant respirators. (11/16/2012 Tr. at 4.17:5-18:6, 4.102:4-103:15; 11/14/2012 Tr. at 2A.77:15-21.) 3M first responded with a technology for making filter material using a fluorochemical melt-additive called PFOS, (*id.*), meaning that fluorine atoms were actually integrated into the melted material (*see* PTX-28.6.). 3M received a patent on this process in 1995, and its employees Marvin Jones and Alan Rousseau were the inventors. (11/16/2012 Tr. at 4.17:8-12, 4.19:2-11; PTX-379.) But in 1998, 3M informed the EPA that there was an environmental problem with PFOS, and in 2000, 3M abandoned PFOS in its products. It was replaced with the melt-additive filtration process, the process that is the subject of this dispute. (*See* 11/16/2012 Tr. at 4.102:8-103:18.)

According to 3M, Jones had successfully "hydrocharged" plasma fluorinated meltblown polypropylene for the first time in August 1995. (11/19/2012 Tr. at 5A.125:19-23; PTX-1143.238-39.) "Hydrocharging" was a 3M patented process invented by Hassan Angadjivand and claimed in U.S. Patent No. 5,496,507 that issued on March 5, 1996 (the "Angadjivand patent"). (DTX-672.) The Angadjivand patent teaches, *inter alia*, "impinging jets of water or a stream of water droplets onto the web at a pressure sufficient to provide the filtration-enhancing electret charge." (*Id.*; *see also* Markman Order at 2, ECF No. 215 ("'hydrocharging' means 'contacting an article with water in a manner sufficient to impart a charge to the article, followed by drying the article'").) Jones and his colleague, Christopher Lyons, continued to experiment

with the hydrocharging process, and eventually achieved a significant increase in the "quality factor"[2] of the material. However, 3M did not initially apply for a patent on the Jones process, to Jones's "chagrin." (11/16/2012 Tr. at 4.12:10-20.)

Eventually, 3M did apply for the patents-in-suit, but not until 1998. (The surrounding saga is told below.) The patents-in-suit claim the method of making an electret article by transferring fluorine to the article from a gaseous phase (the '458 patent), and the method of using fluorinated electrets (the '551 patent). The filtration material—nonwoven, meltblown polypropylene—is made by melting polypropylene[3] pellets in an extruder and blowing the molten polymer through a die, which creates a nonwoven web. (PTX-28.7; 11/14/2012 Tr. at 2A.63:19-64:5.) That material is then charged to create an electret, which improves filtration capacity. (PTX-28.7.) Fluorine atoms are applied to the surface of the web, allowing the filter material to retain its electret in oily environments. (*Id.*)

C. TransWeb's Formation and Patent Applications

In this case, TransWeb asserted that it produced products for public sale using this process over a year before 3M applied for its patents. In 1996, TransWeb was formed to create specialty products for the air filtration market, including fluorinated polymeric nonwoven web. (11/14/2012 Tr. at 2A.40:16-41:5.) On April 30, 1997, Kumar Ogale, a founder of TransWeb, filed a patent application (the '348 application) for an "improved electrostatic filter medium

[2] "Quality factor" is a ratio that relates the pressure drop, which is a measure of the amount of force required for air to pass through the filter, to the effectiveness of the material at removing particles from the air that passes through it. In other words, it balances how well the material filters with how hard a person using the respirator has to breathe through the material. (*See, e.g.*, PTX-28.7 (the '551 patent).)

[3] Polypropylene is a polymer, meaning a long repeating chain of carbon and hydrogen. (11/14/2012 Tr. at 2A.37:19-22.)

including a web of electret fibers which have been treated with fluorine-containing plasma prior to being electrically charged" and "a method of producing non-woven webs which have been treated with fluorine-containing plasma prior to electrostatically charging the web." (PTX-96.6.) That application was rejected in 1998 on the ground that prior art references in the application were already well known to the public, and therefore it would be obvious to a person of ordinary skill in the art to apply fluorine to the filter by plasma treatment. (*See, e.g.*, 11/14/2012 Tr. at 2A.74:8-25.) Ogale and his attorney submitted amendments to the application to the Patent Office, but the examiner issued a Final Rejection on July 9, 1998, because the claims were "unpatentable [under 35 U.S.C. § 103(a)] over the prior art . . . ." (PTX-96.66.) Ogale subsequently abandoned that application. (11/14/2012 Tr. at 2A.74:8-25.)

Ogale later discovered that if he rinsed the fluorinated meltblown polypropylene, it would improve and/or stabilize the electret of the filter material. (PTX-101.) He again applied for a patent, on May 25, 2000, and this time was successful: U.S. Patent No. 6,419,871 (the "'871 patent") issued on July 16, 2002. (*Id.*)

D. Trial

Trial commenced on November 13, 2012, on TransWeb's antitrust and patent declaratory judgment claims, and 3M's patent infringement counterclaims. At the close of TransWeb's case, 3M moved for judgment as a matter of law pursuant to Federal Rule of Civil Procedure 50 on the claims of invalidity, infringement and willfulness, inequitable conduct, and antitrust violations. The Court reserved on those motions.

At the conclusion of the trial, the jury returned a verdict in TransWeb's favor on all but one count, finding that the 3M patent claims asserted were invalid as obvious; that 3M engaged in *Walker Process* fraud in securing and asserting its patents; and that TransWeb did not infringe

3M's patent. Additionally, the jury found that 3M did not commit an antitrust violation by engaging in sham litigation in pursuing its claims against TransWeb. The jury also returned a unanimous advisory verdict of inequitable conduct by 3M,[4] although the Court had instructed the jury that it would accept a non-unanimous verdict on that count. The jury found that TransWeb was entitled to damages in the amount of $34,412 in lost profits plus its attorneys' fees in defending the patent infringement claims, which the jury found were damages attributable to 3M's antitrust violations.

3M then renewed its motions for judgment as a matter of law, and the Court instructed the parties to brief those issues, as well as the inequitable conduct claim, which is for this Court to decide. The Court held a post-trial hearing on January 10, 2013. Issues relating to the damages calculation were submitted to a Special Master, who held extensive hearings during 2013. The Special Master submitted a report and recommendation to this Court on September 24, 2013. This Court then scheduled briefing on objections to the report and recommendation. This Opinion resolves the outstanding post-trial motions; sets forth the Court's findings of facts and conclusions of law with respect to the claim of inequitable conduct; and resolves the objections to the report of the Special Master on damages.

[4] Inequitable conduct was submitted to the jury on an advisory basis at 3M's request.

II. STANDARD ON A MOTION FOR JUDGMENT AS A MATTER OF LAW

A motion for judgment as a matter of law under Fed. R. Civ. P. 50(b) "should be granted only if, viewing the evidence in the light most favorable to the nonmovant and giving it the advantage of every fair and reasonable inference, there is insufficient evidence from which a jury reasonably could find" for the nonmovant.[5] *Lightning Lube, Inc. v. Witco Corp.*, 4 F.3d 1153, 1166 (3d Cir. 1993); *Mandile v. Clark Material Handling Co.*, 131 F. App'x 836, 838 (3d Cir. 2005).

In making this determination, "the court may not weigh the evidence, determine the credibility of the witnesses, or substitute its version of the facts for the jury's version." *Lightning Lube*, 4 F.3d at 1166 (citing *Fineman v. Armstrong World Indus., Inc.*, 980 F.2d 171, 190 (3d Cir. 1992)).

"The question is not whether there is literally no evidence supporting the party against whom the motion is directed but whether there is evidence upon which the jury could properly find a verdict for that party." *Lightning Lube*, 4 F.3d at 1166 (quoting *Patzig v. O'Neil*, 577 F.2d 841, 846 (3d Cir. 1978)).

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[5] Rule 50 provides that, after a party has been heard on an issue at a jury trial, the court may order judgment as a matter of law if it finds that a reasonable jury would not have a legally sufficient evidentiary basis to find for the party on that issue. Fed. R. Civ. P. 50(a). Rule 50(b) involves renewing the motion after trial. In pertinent part, Rule 50(b) provides that: "If the court does not grant a motion for judgment as a matter of law made under Rule 50(a), the court is considered to have submitted the action to the jury subject to the court's later deciding the legal questions raised by the motion." Fed. R. Civ. P. 50(b).

III. JUDGMENT AS A MATTER OF LAW AS TO INVALIDITY-OBVIOUSNESS

3M contends that TransWeb produced insufficient evidence upon which a reasonable jury could find that claims 31 and 57 of the '458 patent were invalid as obvious. The invalidity contention that was submitted to the jury was whether the '458 claims were obvious in light of the purported Ogale prior art product in combination with the Angadjivand patent. The Court will consider 3M's arguments on that issue first, because it bears on a materiality prong of the inequitable conduct allegations.[6]

As the Court instructed the jury, TransWeb had the burden of proving invalidity of the '458 patent by clear and convincing evidence. (Final Jury Instructions at F-7.) *See also* 35 U.S.C. § 282; *Microsoft Corp. v. i4i Limited P'ship*, 131 S. Ct. 2238, 2243 (2011). 3M argues that there was insufficient evidence from which a reasonable jury could conclude that the TransWeb product samples were prior art or that the subject matter of the '458 claims would have been obvious at the time of invention to a person of ordinary skill in the art. *See* 35 U.S.C. §§ 102, 103.

The primary claimed prior art in this case consists of samples of a TransWeb product called T-Melt P, which Ogale testified that he distributed at the 1997 American Filtration & Separations Society Annual Technical Conference and Expo in Minneapolis (the "Minneapolis Expo"). The Expo took place from April 29 to May 2, 1997. (PTX-341; PTX-460.) 3M was a major participant in the Expo, which took place in its hometown, and many 3M employees

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[6] *See Therasense, Inc. v. Becton, Dickinson & Co.*, 649 F.3d 1276, 1292 (Fed. Cir. 2011) ("Often the patentability of a claim will be congruent with the validity determination—if a claim is properly invalidated in district court based on the deliberately withheld reference, then that reference is necessarily material because a finding of invalidity in a district court requires clear and convincing evidence, a higher evidentiary burden than that used in prosecution at the PTO.").

attended it. (*See* PTX-461.22; 11/16/2012 Tr. at 4.34:25-4.35:7.) Ogale testified that he handed out samples of the oil-resistant product and data sheets explaining the filtration properties of the product, including the amount of penetration of the particles and the pressure drop, to visitors to the TransWeb booth at the Minneapolis Expo. (11/14/2012 Tr. at 2A.80:3-88:8.)

A. Public Use

To qualify as prior art pursuant to 35 U.S.C. § 102(b), TransWeb had the burden to prove by clear and convincing evidence that the invention was "in public use or on sale in this country, more than one year prior to the date of the application for patent in the United States." In this case, the '458 patent has an effective filing date of July 2, 1998. (PTX-1314.3.) Therefore, if the jury could conclude that Ogale distributed the T-Melt P samples at the Minneapolis Expo, and/or that he offered them for sale before July 2, 1997, then TransWeb's product would be prior art and the jury's verdict of invalidity rested on ample evidence.

3M contends first that the only evidence that TransWeb displayed and distributed the samples at the Expo is Ogale's testimony. A party must offer more than uncorroborated testimony to carry its burden in invalidating a patent. *See*, *e.g.*, *Finnigan Corp. v. Int'l Trade Comm'n*, 180 F.3d 1354, 1366-67 (Fed. Cir. 1999) (holding that a witness's testimony of an invalidating public use must be corroborated to satisfy the interested party's burden of proving invalidity by clear and convincing evidence). At oral argument on the issue, 3M further contended that TransWeb was obligated to introduce at trial corroborating evidence in the form of corroborating *witness testimony*, and that corroborating documentary evidence was insufficient as a matter of law. Thus, 3M argues, TransWeb was required to put another person on the stand who remembered seeing Ogale distributing those samples at the Expo over 15 years after the Expo ended.

3M controlled the passage of time, and the corresponding diminution of percipient-witness memories by waiting many years before initiating suit.[7] Moreover, the Court finds no support in the law for a requirement that the corroborating evidence be solely a percipient eyewitness rather than another equally valid form of documentary evidence. Notably, 3M provided no cases to support this proposition of law.

Either direct or circumstantial evidence corroborating public use may be sufficient for a party to meet its burden of proof. *See Sandt Tech., Ltd. v. Resco Metal & Plastics Corp.*, 264 F.3d 1344, 1350-51 (Fed. Cir. 2001). The role of circumstantial evidence is particularly important where so much time has elapsed between the key events and the suit. Oral testimony is "more suspect" than contemporaneous documentary evidence, "as there is more of a risk that the witness may have a litigation-inspired motive to corroborate the inventor's testimony, and that the testimony may be inaccurate." *Id.* at 1351. Instead, "each corroboration case must be decided on its own facts with a view to deciding whether the evidence as a whole is persuasive." *Id.* at 1350.

[7] The delay in bringing suit here was caused by 3M's decision not to allege infringement for at least eight years. Prior to trial, 3M claimed that it could not have known whether TransWeb's processes and products infringed its patents because "TransWeb successfully concealed its infringement for years." (3M's Trial Br. 5, ECF No. 433.) The evidence does not support that argument. The evidence shows that 3M specifically sought out TransWeb's products at the Expo (PTX-341, PTX-145, PTX-1336) and years later tested those materials repeatedly (PTX-605, PTX-1336) and even questioned Ogale in multiple meetings about his process and when he had reduced it to practice. (11/14/2012 P.M. Tr. at 60:18-61:11.) Moreover, Hanson, 3M's employee, admitted that he had reviewed TransWeb's '871 patent as soon as it issued in 2002. (11/20/2012 Tr. at 6.9:3-24.) In this litigation, 3M contended that by practicing that '871 patent, TransWeb infringed claims 31 and 57 of the '458 patent. In short, 3M was well aware of TransWeb's alleged infringement by at least 2002. This lawsuit began only after two failed attempts by 3M to purchase TransWeb. (11/14/2012 P.M. Tr. at 61:16-62:19 (2000 attempt); PTX-269 (2008 attempt).)

Where there is corroboration of an interested witness's testimony, the "case questions the sufficiency of the evidence," rather than "the necessity of corroboration," and is therefore appropriately a jury question analyzed under the "rule of reason" test. *Adenta GmbH v. OrthoArm, Inc.*, 501 F.3d 1364, 1371-72 (Fed. Cir. 2007). Here, there was corroborating evidence, as well as Ogale's testimony, upon which a reasonable jury could rely in reaching its verdict. Specifically, there was evidence sufficient for a reasonable jury to conclude that Ogale in fact distributed T-Melt 30P at the Minneapolis Expo in May 1997 and that the product that Ogale distributed at the May 1997 Minneapolis Expo qualified as prior art to 3M's July 1998 patent application.

First, the brochure for the Minneapolis Expo stated that TransWeb would display "T-Melt - Melt blown nonwovens polypropylene" (PTX-460.3), as Ogale testified. (11/14/2012 A.M. Tr. at 80:3-5). Second, 3M's own emails detail how two 3M employees, Mssrs. Sundet and Christensen, were instructed to make contact with TransWeb at the Expo and discuss their "T-Melt" product. (PTX-341.) After the Expo, Christensen stated that he and Sundet had picked up samples of the T-Melt product, which would be shipped to 3M, and included the price range for the T-Melt material. (PTX-46.2.) Based on 3M's own documents, it can scarcely be ingenuously claimed there is no corroboration for TransWeb's assertion that Ogale handed out samples of the product at the Expo.

Third, Ogale's patent application describing the T-Melt P product was filed during the Expo. (PTX-96.) That application described the process that TransWeb attempted to patent, which produced the prior art that Ogale testified he distributed. Fourth, TransWeb introduced evidence consisting of correspondence between itself and a company called Fourth State, to which TransWeb initially outsourced its fluorination, setting the fluorination conditions for the

creation of the oil-resistant product distributed at the Expo. (PTX-84; PTX-86.) Fifth, TransWeb introduced documentary evidence that it was providing samples of the same material to potential customers, including Juergen Binzer, MSA, Filtration Group, and Racal. (PTX-1345; *see also* 11/14/2012 A.M. Tr. at 79:8-16, 89:16-92:2; PTX-82.19 (documenting sending of samples to MSA and Racal); PTX-441 (letter from MSA returning samples); PTX-102 (letter to Filtration Group); PTX-75 (shipping documents reflecting shipping of samples from TransWeb to Racal on May 8, 1997)). Sixth, a letter from TransWeb to the Louis M. Gerson Co. on July 1, 1997—shortly after the Minneapolis Expo—demonstrates that TransWeb was prepared to provide the prior art product to a purchaser around the time of the Expo (and before the 3M priority date). (PTX-61.)

Finally, TransWeb introduced a physical exhibit that the jury could reasonably find was a set of the actual sheets of the TransWeb T-Melt 30P product with a data sheet describing its performance. (PTX-1338.) That exhibit was found in the files of 3M employee Pierre Legare. (PTX-1353.) Just a few months before trial, when it first appeared on the proposed exhibit list for 3M, it had been identified by 3M's outside counsel only as "a physical sample comprised of four sheets of material" (*id.*), with no description connecting it to TransWeb nor identifying it by name, nor when or how it came into the possession of Legare and 3M. As the intense dispute over this piece of evidence emerged at the trial, it became clear that this identification of the evidence may have been deliberately opaque, which set in motion a drama (outside the presence of the jury).

Shortly before the Final Pretrial Conference, 3M disclosed for the first time that the Exhibit, PTX-1338, was a "TMelt 30P Sample." In other words, it was TransWeb's precise product and was key evidence—perhaps even "killer" evidence—of what TransWeb contended

was invalidating prior art. (*Id.*) Thus, the date when Legare or 3M obtained PTX-1338 was a fact of no minor importance. That sample—from 3M's own files—exactly matched Ogale's description of the samples that he distributed at the May 1997 Minneapolis Expo, which would show public use and distribution more than one year before 3M's July 1998 effective filing date for its patent. (11/14/2012 Tr. at 2A.83:25-84:16.) By contrast, Defendants claim that the PTX-1338 sample was received under a confidentiality agreement on June 2, 1997 when Ogale visited Racal (where Legare then worked). Thus, Defendants argue that PTX-1338 was not publically distributed at the Minneapolis Expo in May 1997; that it was instead received by Legare under a confidentiality agreement and then sent to 3M for testing; and that it is thus not evidence of public distribution more than one year before the July 2, 1998 effective filing date of 3M's patent.

But the sample that TransWeb provided to Legare had been sent to Jones at 3M for oily testing. Jones testified that he did conduct a test on the sample that Legare had sent him—which required putting drops of oil on the sample. (11/19/2012 Tr. at 5A.63:15-20.) If Defendants' theory about the manner and date of receipt of PTX-1338 were true, then the samples would be soiled by the oily testing. PTX-1338, however, are sheets in pristine condition. This evidence also supports the jury verdict and indicates that PTX-1338 was a sample distributed at the Expo in May 1997 and taken back to 3M, and that it was not the sample delivered to Legare at the June 1997 meeting. Based on the condition of PTX-1338, the jury could well have found that this sample packet was either picked up at the Expo or mailed to Legare immediately at the conclusion of the Minneapolis Expo—cut from the same material that Ogale testified that he took to the Expo (11/14/2012 Tr. at 2A.84:22-85:17, 2A.95:13-16, 2A.96:9-97:10)—or that

PTX-1338 was picked up by Legare's coworker, Suresh Kalatoor, who attended the Expo, where Ogale was handing out these samples in this identical type of packaging. (PTX-461.22.)

In short, "this is not a case where one person makes a naked, unsupported assertion years after the fact that he made an invention before a patentee." *Adenta*, 501 F.3d at 1371. Here, Ogale's testimony was "accompanied by various supportive and consistent documents," which, viewed in light of the amount of time that has elapsed since the events, "provided a coherent and convincing story." *Id.* That corroboration is sufficient to guard against the possibility of "fraud, by providing independent confirmation of the inventor's testimony." *Kridl v. McCormick*, 105 F.3d 1446, 1450 (Fed. Cir. 1997).

B. Whether the Method Can Be Determined

For the Expo samples to qualify as prior art against 3M's patented method, a person of ordinary skill in the art must be able to determine TransWeb's method from analyzing these samples. *See W.L. Gore & Assocs., Inc. v. Garlock, Inc.*, 721 F.2d 1540, 1550 (Fed. Cir. 1983) (holding that, to be prior art against the method claimed in a patent, sales of a physical product require "evidence[] that the public could learn the claimed process by examining the [physical product]"). Here, there was sufficient evidence upon which the jury could rely in reaching that conclusion.

Significantly, there was sufficient evidence from which the jury could have well inferred that 3M's employees *did in fact* determine TransWeb's method from analyzing these (or nearly identical) samples. In a report dated December 2, 1998, 3M's analysis of the TransWeb product revealed that the T-Melt 30P material was "a non-woven web"; that the surface of that web contained between 51% and 47% atomic fluorine; and that the web exhibited a carbon spectra comprising "several ove[r]lapping peaks indicative of the multiple states formed by exposure to

a high energy plasma or corona type treatment." (PTX-29.6-7.) Relying on that report, 3M's later Supplemental Information Disclosure Statement to the PTO stated about the T-Melt 30P product that it was a nonwoven, polymeric filtration web that was surface fluorinated with a CF3:CF2 ratio of at least 0.45, and possibly greater than 0.9, a quality factor between 0.13 and 0.62, and a basis weight of 30 g/m^2. (PTX-29.1.)

Other documents and testimony from 3M employees confirm that the method could be readily determined from the samples. For instance, Legare wrote in an email to Jones that the Racal samples were "samples of 30g and 50g charged web from TransWeb" that were "corona charged polypro BMF with fluoropolymer treatment." (PTX-47.) Similarly, in his email to Ogale, Kurtzahn, the 3M Procurement Manager, stated that none of the samples "we are receiving from you is confidential," and that if 3M "wished to duplicate your product or litigate, we would not have to request samples from you," because 3M could "obtain the product in the marketplace." (PTX-26.)

TransWeb's expert Dr. Edward Funk also testified that one of ordinary skill in the art could determine how TransWeb made its samples by examining and feeling the samples and running Electron Spectroscopy for Chemical Analysis tests to determine fluorination, and DOP penetration tests before and after exposing the material to radiation to determine the charge. (11/20/2012 Tr. at 207:9-23; 207:24-208:3; 209:4-210:6; 210:12-211:1.) The jury was entitled to give this testimony weight in rendering its verdict.

The Court will therefore deny 3M's Motion for Judgment as a Matter of Law on the above-described grounds.

IV. INEQUITABLE CONDUCT

Inequitable conduct is the "atomic bomb" of patent litigation, because it renders an entire patent—and in this case an entire patent family—unenforceable. *Therasense, Inc. v Becton, Dickinson & Co.*, 649 F.3d 1276, 1288 (Fed. Cir. 2011) (quoting *Aventis Pharma S.A. v. Amphastar Pharm., Inc.*, 525 F.3d 1334, 1349 (Fed. Cir. 2008) (Rader, J., dissenting)). The Federal Circuit has expressed its concern at the large number of cases in which defendants allege inequitable conduct by the patentee, commenting that it "has plagued not only the courts but also the entire patent system," by nearly requiring cautious patent prosecutors to "bury PTO examiners with a deluge of prior art references." *Therasense*, 649 F.3d at 1289. This Court has long shared the Federal Circuit's concern. *See, e.g., Fuji Photo Film Co. Ltd. v. Jazz Photo Corp. Inc.*, 173 F. Supp. 2d 268, 276 (D.N.J. 2001) (concluding that there is "no clear and convincing evidence of inequitable conduct" and granting summary judgment); *Teva Women's Health, Inc. v. Lupin, Ltd.*, Civ. No. 10-80, 2010 WL 4392503, at *1 (D.N.J. Oct. 27, 2010) (dismissing claims for inequitable conduct).

The PTO imposes on practitioners who apply for patents a duty to disclose information material to patentability. 37 C.F.R. § 1.56. The duty applies to "[e]ach individual associated with the filing and prosecution of a patent application," *id.*, and expressly to "each inventor named in the application" and "each attorney or agent who prepares or prosecutes the application," as well as "every other person who is substantively involved in the preparation or prosecution of the application and who is associated with the inventor, the applicant, an assignee, or anyone to whom there is an obligation to assign the application." *Id.* at 1.56(c). The PTO depends on their honest and forthcoming disclosure of material prior art references of which they are aware

because "the most effective patent examination," requires the Examiner to be "aware of and evaluate[] the teachings of all information material to patentability." *Id.*

Therasense sets a very high bar for the accused infringer who seeks to defend on this ground: "the accused infringer must prove by clear and convincing evidence that the applicant knew of the reference, knew that it was material, and made a deliberate decision to withhold it." *Therasense*, 649 F.3d at 1290.

A. Materiality

In *Therasense,* the Federal Circuit held that, except in cases of "egregious misconduct," the accused infringer must show "but-for materiality," meaning that "the PTO would not have allowed a claim had it been aware of the undisclosed prior art." *Therasense*, 649 F.3d at 1291-92 (rejecting the "low bar for materiality" set in PTO Rule 56.)

In this case, the materiality of the TransWeb prior art is clear. When it was disclosed to the examiner, the examiner rejected the claims in 3M's patent application as obvious based on TransWeb's prior art combined with the Angadjivand patent—the same conclusion reached by the jury. (PTX-21.4 ("Consequently, it would have been readily obvious to one having ordinary skill in the art to employ the above-noted expedients in the TW filter web in order to facilitate the removal of undesirable contaminants as taught by Angadjivand et al ('507).").)[8] To reverse the PTO's rejection, 3M argued to the Examiner that the TransWeb prior art was *only* available under a confidentiality agreement (PTX-22). The Examiner then withdrew the rejection of the claims based on that representation alone. (PTX-1312.321 ("In light of applicants' arguments filed on May 14, 2001, the rejection of all the pending claims 2-7, 21, 22, 45, and 52-59 as

[8] In fact, 3M's brief does not appear to challenge the but-for materiality of the Expo samples, assuming they are prior art. Instead, 3M contends that they are not qualifying prior art as a matter of law. For the reasons set forth above, that is not correct.

applied in paragraph 4 on page 3 of the last Office action (Paper No. 27) dated January 29, 2001 [PTX-21.4] has been withdrawn.").) The prior art, if public, would invalidate the claims asserted, as the jury found. *See Therasense*, 649 F.3d at 1292 ("[I]f a claim is properly invalidated in district court based on the deliberately withheld reference, then that reference is necessarily material because a finding of invalidity in a district court requires . . . a higher evidentiary burden than that used in prosecution at the PTO.").

In sum, 3M's in-house counsel and inventor employees told the Examiner *only* about the Racal samples: these samples, they argued, were covered by a confidentiality agreement.[9] 3M's disclosure completely ignored the earlier Minneapolis Expo samples that Ogale had publicly distributed at the Expo, under 3M's careful, pre-planned focus at that Expo, as the facts adduced at trial showed beyond peradventure.

The Court now turns to the issues of knowledge and intent.[10]

B. Knowledge/Intent

As stated above, *Therasense* sets a heavy burden to prove knowledge and intent. In addition to clear and convincing evidence that the applicant knew of the reference, knew that it was material, and made a deliberate decision to withhold it, there must also be proof that the patentee acted with specific intent to deceive the PTO. 649 F.3d at 1290 (citing *Star Scientific Inc. v. R.J. Reynolds Tobacco Co.*, 537 F.3d 1357, 1366 (Fed. Cir. 2008)). "Because direct evidence of deceptive intent is rare, a district court may infer intent from indirect and circumstantial evidence." *Id.* (citing *Larson Mfg. Co. of S.D., Inc. v. Aluminart Prods. Ltd.*, 559

[9] In fact, even these samples were sent to Racal without a confidentiality agreement in place, and only later was a confidentiality agreement entered into between TransWeb and Racal.

[10] "Intent and materiality are separate requirements." *Therasense*, 649 F.3d at 1290 (rejecting the "sliding scale" for intent and materiality that prevailed previously).

F.3d 1317, 1340 (Fed. Cir. 2009)). However, specific intent to deceive "must be the 'single most reasonable inference able to be drawn from the evidence.'" *Id.* (quoting *Star*, 537 F.3d at 1366.) Accordingly, "when there are multiple reasonable inferences that may be drawn, intent to deceive cannot be found." *Id.* (citing *Scanner Techs. Corp. v. ICOS Vision Sys. Corp.*, 528 F.3d 1365, 1376 (Fed. Cir. 2008).)

Here, TransWeb alleges that Jones, a 3M inventor, and Hanson, a 3M in-house patent prosecutor, engaged in inequitable conduct during the prosecution of the application that became the '458 and '551 patents. Both owed duties of candor to the PTO under the express language of PTO Rule 56. The question is whether they knowingly and deliberately withheld the reference to the Expo samples (and only disclosed the purportedly confidential Racal samples) with specific intent to deceive the PTO and thereby reverse the rejection of the patent based on these samples. If so, is specific intent to deceive the single most reasonable inference to be drawn from the evidence?

The evidence established that 3M inventors and employees, including Jones and Rousseau, were well aware of TransWeb and its products prior to and during the Minneapolis Expo. As stated above, the evidence showed that high-level 3M employees, acting pursuant to an advance plan, targeted the TransWeb booth at the Expo. The day the Expo began, Colleen Nagel, Jones's and Rousseau's supervisor, sent an email to Jones and Rousseau, reminding them again about her interest in TransWeb's product, because attached to it she re-sent them an email she had earlier sent, which reprinted a TransWeb press release from the April 1997 edition of the trade magazine *Nonwovens Industry*. (PTX-341.) The re-sent email, entitled "Transweb," noted TransWeb's new "electret fiber nonwoven line" producing the "T-Melt" product, targeting, among other things, the respirator market. (*Id.* at 341.2.) The email also essentially instructed

Jones and others that, in order to avoid too many 3M employees from engaging in conversation with TransWeb, lest it overtly show too much interest in TransWeb, the oral contacts between 3M and TransWeb would be Doug Sundet and Curt Christensen, who were designated the 3M "point people in [3M's] discussions with Transweb." (*Id.*) The email asked Jones and Rousseau, and the other recipients, to let Sundet and Christensen know if they would like information from TransWeb, including samples of TransWeb material. (*Id.*) The implication of the email is clear: 3M was highly interested in TransWeb's products. "Look, but don't engage in conversation," was the clear message to Jones and Rousseau; the talking would be done by Sundet and Christensen to avoid too many 3M employees showing TransWeb how interested they were in what it was distributing.

Jones did in fact attend the Expo, as did numerous other 3M employees (PTX-461.22), and his testimony about the Expo is revealing, especially in the context of the repeated emails from his boss, who was very interested in TransWeb. He recalled with impressive detail certain aspects of the Expo—for instance, describing a company producing meltblown material in the exhibition hall of the Expo, and recounting his interest in a company called TetraTech, which had a booth right next to TransWeb's booth at the Expo, and which also made oil-resistant filter material. (PTX-460; 11/16/2012 Tr. at 4.47:21-4.52:7.) Yet Jones professed not to remember any details about whether he visited or even saw the TransWeb booth, and changed his explanation of whether he even went to the floor of the Expo, where vendors like TransWeb were located.[11]

[11] At one point, Jones stated clearly "I was not in the expo area." (11/16/2012 Tr. at 4.50:6.) Merely two questions later, when TransWeb's counsel asked him to confirm that he had not walked by TransWeb's booth in the expo area, he responded, "No, that's not what I testified. I have no idea . . . . I cannot say that it's impossible." (*Id.* at 4.50:12-14.) Later, he wavered repeatedly about whether he had visited the TetraTech booth immediately adjacent to the TransWeb booth. He indicated he "was more interested" in TetraTech than TransWeb (*id.* at

(*See* 11/16/2012 Tr. at 4.47:21-4.52:7.) His testimony about his experience at the Expo (and other areas of his testimony) was simply not credible, particularly in light of the emails, exhibits, and his hostile, evasive, and sometimes flippant or arrogant demeanor. In short, his demeanor was one of a witness trying to distance himself from any knowledge of TransWeb's presence and activities at the Expo. While his boss said to him, in essence, "look, watch, figure out what you want from his booth, but don't chat directly because Sundet and Christensen are doing that." He pretended that he barely knew, or perhaps didn't even know, that TransWeb was there. In short, his testimony was not credible, and at times was hard to watch.[12]

Shortly after the Expo, on May 27, 1997, Jones's boss, Colleen Nagel, again sent Jones an email about TransWeb,[13] this time writing "fyi relevant to your transweb meeting."[14] (PTX-46.1.) The email she forwarded with that message was an update from Christensen, 3M's "point

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4.48:23-24), but that he "doubt[ed]" he went to the TetraTech booth (*id.* at 4.49:3), and then later that he was certain he would not have been interested in any oil-resistant material at the TransWeb booth because "they had [oil-resistant media] at the adjoining booth"—the TetraTech booth—in which he was more interested (*id.* at 4.49:23-4:51-1.). Moreover, at trial, Jones was confronted with multiple brochures distributed by exhibitors at the Expo, which were produced to TransWeb from his files shortly before trial began. (*Id.* at 4.42:6-4.46:17.) He could not explain how he received the brochures. (*Id.*).

[12] Trial Counsel was not to blame for any of this; they did the best they could with a very difficult witness who was called by the other side early in the case.

[13] Attempting to explain his inability to recall Nagel's emails about TransWeb, Jones stated that he would only have read an email from his manager "[i]f I saw it," and that he was "not a hundred percent reliable" when it came to reading email from his boss. (11/16/2012 Tr. at 4.60:1-6.)

[14] The evidence shows that during this time period, Ogale and other TransWeb employees were making sales calls to potential purchasers of T-Melt P materials. For instance, during this time period, Ogale visited Legare at Racal, and at some time provided samples of the T-Melt P material. (*See generally* 11/26/2012 P.M. Tr. at 134:20-135:9.)

person" for TransWeb, recounting what he had learned from TransWeb at the Expo, including that TransWeb's "Meltblown and Electret (fibrillated PP) production lines are up and running essentially on schedule." (PTX-46.2.) Christensen's email also stated that he and Sundet had picked up samples of TransWeb's material at the Expo, and that additional material was being shipped to 3M, and even included price ranges for the material. (*Id.*) Jones professed to have no recollection of any meeting with TransWeb.[15]

TransWeb again came to the attention of Jones and Rousseau during the summer of 1998. 3M had purchased Racal in March 1998, and Legare and Suresh Kalatoor of Racal were by then 3M employees. Legare and Kalatoor visited Minneapolis and attended a barbeque with Jones at Rousseau's house. (11/27/2012 Tr. at 8A.31:17-32:12.) While he was there, Legare told Jones and Rousseau that he had received samples of TransWeb oil-resistant product for respirators, and, according to Legare, they responded: "That's interesting." (*Id.* at 8A.33:22-35:8; 8A.36:25.) Ogale testified that he had also already told Legare that he was applying for a patent for his process, which involved surface fluorination (11/14/2012 Tr. at 2A.97:11-25), but Legare claimed that he could not recall that at trial. (11/27/2012 Tr. at 8A.30:8-11.)

After years sitting dormant, 3M then began to rush its patent application. Christopher Lyons, one of Jones's co-inventors, admitted that the filing was rushed. (11/27/2012 P.M. Tr. at 40:19-42:4; 50:1-12.) Indeed, for the only time in all of the years covered by Lyons's ordinarily impeccably organized and continuously and contemporaneously witnessed lab notebook, Lyons

[15] In fact, Jones stated that he had no recollection of having ever seen Ogale before the trial. (11/19/2012 P.M. Tr. at 31:20-32:2.) However, Hanson testified that just one year ago, Jones told him that he met Ogale for the first time in 1997 or 1998. (11/20/2012 Tr. at 6.10:18-12:8.)

kept imperfect notes that were not witnessed[16] from that summer of 1998 until December 1998, when 3M tested the TransWeb T-Melt 30P material it received from Racal. (*Id.* at 47:5-48:9.) On December 4, 1998, two days after the TransWeb T-Melt 30P sample had been analyzed by 3M, Lyons went back and entered all of his entries for that six-month period, including work that was purportedly done related to the patent application filed six months earlier on July 2, 1998, which was not "witnessed" until December 28, 1998. (PTX-1130.37-42.) Many of the examples in the specifications of the '458 and '551 patents were performed within weeks of the application filings (PTX-1125.112-120), even though 3M had purportedly been working on the inventions for over a decade, and the inventors did not sign their declarations until six weeks after the application was filed. (PTX-1312.37-38.)[17].

On November 11, 1998, 3M employees tested a Willson P-95 respirator, as requested by Jones and Rousseau. (PTX-610 (November 11, 1998 email attaching withheld ESCA testing results for Willson P-95); PTX-605 (December 2, 1998 email from Michael Prokosch to Jones and Rousseau and a third individual referring to "the Willson P-95 sample that [they] had recently submitted").)[18] On that date, if not before, Jones and Rousseau knew that plasma-fluorinated oil-resistant respirator media—the subject matter of their pending patent

16 3M procedures required inventors to describe their work daily in their lab notebooks and to have witnesses corroborate the notebook entries. (PTX-1130.3-5.)

17 Jones's notebook was consistently flawed. The entries allegedly from June 1997 until July 1, 1998, were all witnessed on July 8, 1998. (PTX-1125; 11/19/2012 Tr. at 5A.85:10-19.)

18 Jones confirmed that, in his experience, from the date an application for NIOSH certification is made to actually getting a respirator product on the market could take months. (11/19/2012 Tr. at 5A.26:23-27:2.) The clear inference is that the Willson P-95 respirator had been on the market for quite a while.

application—was on the market. The clear inference from the evidence is that they also had reason to know that the filter material in that respirator was the TransWeb material. One week later, at Rousseau's request, Legare mailed the Racal samples of TransWeb material to Jones, which he confirmed in an email to Jones, copying Rousseau and Nagel, with the subject line "Transweb." (PTX-47 (identifying the samples as fluoropolymer corona charged polypropylene meltblown fiber); 11/19/2012 Tr. at 5A.38:22-5A.39-12.)[19] 3M tested the Racal samples of TransWeb's T-Melt 30P product, and a report analyzing them was circulated to Jones and Rousseau on December 2, 1998. (PTX-605.) In the email accompanying that report, Michael Prokosch of 3M noted that the TransWeb sample was "quite similar" to the Willson P-95 respirator's filter. (*Id.*) Jones then called Legare, who stated of the TransWeb sample: "of course they were received under a . . . confidentiality agreement," something Legare had never previously said to Jones or Rousseau, nor was any mention of it made when Legare sent the TransWeb samples to Jones. (11/19/2012 Tr. at 5A.73:21-5A.74:8; 5A.108:8-9.)

Clearly, a conversation then ensued within 3M about TransWeb's product that had been distributed the prior year at the Minneapolis Expo. A little over three weeks after testing the Willson respirator that contained the TransWeb material, on December 27, 1998, Rousseau forwarded to Jones and patent attorney Hanson the email from the start of the 1997 Expo that Nagel had earlier sent about TransWeb. (PTX-341.) (That was the email that notified Jones, Rousseau, and others that TransWeb would exhibit at the Expo a new "electret fiber nonwoven line" producing the "T-Melt" product targeting, among other things, the respirator market. (*Id.*))

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[19] In his email to Jones and Nagel, Legare made no mention of any confidentiality agreement with TransWeb, and at trial he could not explain why he would send confidential samples to 3M, TransWeb's primary competitor. (11/27/2012 Tr. at 8A.42:3-44:12.)

Forwarding the email to Jones and Hanson, Rousseau stated, "Found this while cleaning out old notes. Notice that at this time there was no mention of oil resistant electret filter web." (*Id.*)[20] The inference is clear that Rousseau realized that the TransWeb products at the Expo and in *Nonwovens Industry* were likely the same products 3M had just analyzed and found in the Willson respirator. And thus he so informed Jones and Hanson.[21] The comment about "no mention of oil resistan[ce]" appears to be a follow-up to an oral conversation they had been having about the TransWeb product given out at the Expo over a year earlier.

Recognizing the problem, Jones and Rousseau and attorney Hanson spoke with Gary Kurtzahn, a purchasing manager, and instructed him to order samples from TransWeb. (PTX-1336 ("I requested the various samples from Transweb on 3/15/99 per our telecon.").) In his request to TransWeb, Kurtzahn noted that the materials had been advertised in *Nonwovens Industry* (PTX-145)—indicating that they were advertised more than one year before the 3M priority date—and clearly referring to the email Rousseau and Jones received from Colleen Nagel before the 1997 Expo.[22] Sometime during the correspondence with Kurtzahn in 1999, a

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[20] Despite the fact that he knew that 3M's Sundet and Christensen had been the point men for collecting information and samples from TransWeb, Hanson testified that he never asked Sundet or Christensen about the Expo. (11/19/2012 P.M. Tr. at 111:3-17; 11/20/2012 Tr. at 6.83:1-84:2.)

[21] Indeed, there is no evidence to suggest that as of December 27, 1998, Jones, Rousseau or Hanson were aware of any non-fluorinated T-Melt product made by TransWeb.

[22] In its brief, 3M argues that Kurtzahn's request for the samples was "in order to consider it as a potential supplier of filter media for several 3M businesses." (3M's Br. in Supp. of its Request for a Finding of No Inequitable Conduct 6, ECF No. 516.) That is not a plausible explanation in the context of other contemporaneous conversations between the inventors at 3M, as well as the Willson testing, all occurring at 3M at that time, nor in the context of the emails preceding the letter, nor the letter itself, which refers back to the April 1997 edition of *Nonwovens Industry*. Moreover, the individuals who asked Kurtzahn to order the samples were the two inventors and the patent prosecutor for the 3M patents-in-suit. The only reasonable

confidentiality agreement provided by 3M was signed by Ogale. (PTX-25.) The samples were then delivered to Jones, although Jones professed to have no memory of receiving them. (11/19/2012 Tr. at 5A.82:5-24.)

Meanwhile, Hanson—the patent prosecutor, who at that time had never contacted TransWeb—proceeded to write to Ogale a letter which a non-attorney would read as being about 3M's interest in doing business with TransWeb in 1999, but which interwove in its verbiage a description of a purported prior confidentiality agreement between 3M and TransWeb. At the end of the letter, attorney Hanson asks Ogale to confirm its contents, which has this rather odd interlineation of a reference to a confidentiality agreement mixed in with a 1999 transaction. This Court still cannot discern a good faith purpose of the interlineation in that letter, and 3M has struggled mightily to explain it. The letter clearly asks Ogale to agree to the termination of the confidentiality agreement between Racal and TransWeb, which was signed before Racal was ever acquired by 3M, and then goes on to say that the 1999 samples sent by TransWeb to 3M are covered only by the 3M confidentiality agreement provided by Kurtzahn. (PTX-641.) However, Hanson also intermixed in this letter a few unnecessary extraneous lines stating that TransWeb had provided Racal with samples of its T-Melt products pursuant to the June 2, 1997 TransWeb-Racal confidentiality agreement, when in fact samples were sent before that date without a confidentiality agreement.[23] (*Id.*) Ogale signed that letter, which was most easily understood by a

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inference is that Kurtzahn's request was because of the concern of these 3M employees that TransWeb's products were prior art.

[23] Legare testified that all of the T-Melt P samples that Racal received were delivered by Ogale on June 2, 1997, and therefore covered by the Racal-TransWeb confidentiality agreement signed at that meeting. Despite purportedly recalling that meeting in great detail, he had no recollection of a meeting he had with Ogale just six months before, in late 1996. (PTX-72; 11/26/2012 P.M. Tr. at 128:12-129:6.) He also could not explain which samples were sent to him

businessman as confirming the 1999 confidentiality agreement. However, Hanson and 3M continually quote only the oddly intermixed lines as an "admission" by Ogale that the 1997 samples were given to Legare on June 2, 1997, when a confidentiality agreement was signed. (*Id*.) And when Hanson finally disclosed the Racal samples to the PTO, he represented that Ogale agreed that they were covered by the TransWeb-Racal confidentiality agreement—and said nothing more about the actual subject of the letter. (PTX-29.1) No reasonable patent examiner, juror, or judge could find that 3M's selective quotation and proposed interpretation of the letter makes much sense to lawyers, let alone lay businessmen. It is clear on the letter's face that Ogale would have thought that he was agreeing that the new samples were subject to the 3M-TransWeb agreement, and that the Racal-TransWeb agreement had ended. Hanson wrote that letter in 1999, ostensibly about the 1999 confidentiality agreement, as a pretext to get a purported "admission" about the timing of the earlier TransWeb-Racal interaction. In fact, this Court is persuaded by the evidence that the samples were shipped to Racal prior to the signing of any confidentiality agreement between Racal and TransWeb.

Hanson apparently discussed with Legare whether the Racal samples were public. In 1997, right after the Expo, Ogale had mailed the samples to Racal without any request for a confidentiality agreement, and thereafter when Legare and Ogale met in person, a confidentiality agreement was signed at Legare's office at Racal. (*See* n.23.) Once Hanson began to focus on the problem, he met with Legare, who only then typed up the handwritten notes he had taken in his meeting with Ogale some eighteen months prior. (11/27/2012 Tr. at 8A.22:9-23; *compare* PTX-

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by Ogale in May, one month before the June 2 meeting. (PTX-75.2-4.; 11/26/2012 P.M. Tr. at 135:10-13.) Finally, Legare's notes implied that he did not in fact receive the samples at the June 2 meeting. (DTX-153.)

90 (the typed notes) *with* DTX-153.) The inference is clear that Hanson asked for a typed version of the notes. The typed notes have additional information that was not included in the handwritten contemporaneous notes—for instance, that "discussion ensued around 3M patent, Kumar assured no conflict with 3M as different technical approach applied but no details provided;" and "My comment Racal does not want to be pulled into any actions from 3M so they need to be absolutely certain of their position." (PTX-90.) Legare could not explain why those statements were not in the original notes. (*See* 11/27/2012 Tr. at 8A.26:1-11, 27:18-28:7.) Hanson's meeting with Legare thus led to materially altered meeting notes, and is circumstantial evidence of the false Legare story about the origin of the PTX-1338 samples. Hanson helped prepare Legare for his deposition, and attended Legare's deposition (11/26/2012 P.M. Tr. at 141:16-19). And then we have Legare's surprise—dubious—assertion that the TransWeb sample pack marked PTX-1338 was provided by Ogale to Racal on the day Ogale signed a confidentiality agreement, which even 3M's own outside counsel was "unable to confirm" after a lengthy "investigation." (PTX-1353.)

Legare's testimony was not credible in crucial respects, particularly in his manner when challenged about his sudden new recollection at trial of a precise date when he received PTX-1338. For the first time, 15 years after the event, when no dates appeared on PTX-1338 nor any associated document, Legare testified to a precise, self-serving date for when he got the sample pack. Indeed, evidence was introduced showing that 3M's own lawyers did not believe Legare. When 3M finally disclosed, just before Final Pretrial Conference, that PTX-1338 was a TransWeb product sample found in Legare's files, a testy email correspondence between TransWeb's counsel and one senior associate on 3M's trial team ensued, in which TransWeb counsel asked how and when Legare acquired the samples. (PTX-1353.) That senior associate

represented that 3M was "investigating" that issue, and also that TransWeb was not entitled to the information. (*Id*.) When TransWeb threatened to raise the issue with the Magistrate Judge at that Final Pretrial Conference and repeatedly asked for more information, the senior associate stated that the matter was still being investigated. When pressed for the fourth time about the results of 3M's "investigation," the senior associate[24] represented that "3M ha[d] been unable to confirm when Legare obtained this sample." (*Id*.)

The senior associate's representation ensured that TransWeb would not raise the discovery issue with the Magistrate Judge and seek to reopen Legare's deposition, a disturbing tactical maneuver on professionalism grounds.[25] But, as questionable tactics often do, it also boomeranged as a non-privileged substantive admission of 3M that effectively impeached its own witness' sudden resurrected memory of that date. Legare did in fact testify at trial that he received the sample at the June 2, 1997 meeting. This incredible testimony by Legare formed the basis of 3M's contention that the PTX-1338 samples—exemplars of the prior art withheld from the PTO—were covered by the confidentiality agreement of that date. Legare also testified that he "absolutely . . . told [3M counsel] that I had gotten it at that meeting, as I've told everyone that I got it at that meeting." (11/27/2012 Tr. at 8A.19:9-11.) The only reasonable interpretation of counsel's statement after so much "investigation"—that 3M could not confirm when Legare received the sample—is that 3M's own counsel doubted his statement.

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[24] No other attorney on 3M's trial team apart from this particular senior associate was aware of this side drama until it arose at trial.

[25] While not relied upon for purposes of the findings in this opinion, the senior associate on 3M's trial team actually *knew* when she sent the email, but did not disclose, that Legare would testify at trial that he received the sample on that key date of June 2, 1997, even though months of "investigation" could not confirm the accuracy of this date of receipt.

There was good reason to doubt the credibility of Legare's testimony about this important date. The facts are, instead, that samples were shipped by TransWeb to Racal before June 2, with no request for confidentiality. On June 2, at Legare's request, a confidentiality agreement was executed. These samples were later sent to Jones for oily testing and would thus be dirty; and PTX-1338 is untouched by oil and in pristine condition. PTX-1338 is a TransWeb sample from Legare's files that looks just like the samples publicly distributed by Ogale at the Expo (and immediately thereafter).

Legare's credibility problems were compounded by his selective memory. When describing the dates that key events occurred, without fail, he purported to remember in great detail dates which helped 3M's case (such as the June 2 meeting), but he could not recall any other details, including purportedly forgetting entirely a meeting he had with Ogale a mere six months prior (PTX-72; 11/26/2012 P.M. Tr. at 128:12-129:6), or receiving a mailed sample of the filtration material from TransWeb in May 1997, prior to the June meeting. (PTX-75.2-4.; 11/26/2012 P.M. Tr. at 135:10-13.) Legare's self-serving selective memory is unsurprising, as he met with Hanson in the months leading up to trial (11/26/2012 P.M. Tr. at 143:3-9; 11/27/2012 Tr. at 8A.9:15-19), and, contrary to 3M's representations, they did not discuss any other patents during those meetings (11/27/2012 Tr. at 8A.53:8-10). They were thus discussing this case.

Regarding Hanson's conduct with respect to the PTX-1338 samples, he waited many years before he disclosed anything about TransWeb's prior art product to the Examiner. Instead, 3M first explored the possibility of purchasing TransWeb, while continuing to probe information from Ogale about the prior art. On April 20, 2000, 3M invited Ogale to visit 3M headquarters, and 3M employees asked him for information about TransWeb's business and product lines, including how long the company had been in business. (11/14/2012 P.M. Tr. at 60:18-61:11.)

Following the meeting, John Reed, a 3M employee, circulated an email to numerous 3M employees including Rousseau and Legare, in which he wrote, "As a follow-up note to the meeting on April 20 with Transweb . . . Minimize direct contact with TransWeb. . . . Minimize written exchanges. Maximize verbal exchange of info . . . ." (PTX-1335.) The clear intent was to grill Ogale about the prior art, under the pretext of 3M becoming a big customer of TransWeb. In July 2000, 3M invited Ogale to return to Minneapolis with Richard Granville, his angel investor, and expressed its interest in purchasing the entire TransWeb company. (11/14/2012 P.M. Tr. at 61:16-62:19.) Those discussions fell apart. (*Id.* at 62:21-22.)

Only after the acquisition discussions failed, upon being notified that 3M's patent was ready to issue, did Hanson finally make a Supplemental Information Disclosure to the Examiner. (PTX-29.) That disclosure caused the Examiner to reject 3M's patent. While there may be no violation of the duty of disclosure where the information is before the examiner in time for him to act on it, *Young v. Lumenis, Inc.*, 492 F.3d 1336, 1349 (Fed. Cir. 2007), the law does not authorize deliberate strategic withholding of information, nor does it permit the applicant to choose to delay disclosure to suit its own timetable for issuance of the patent. Here, however, strategic delay is not the only problem. The real problem is that the substance of what was finally disclosed was not the whole truth. Hanson attempted to avoid disclosure for as long as possible, and, when that could no longer work, he and others deceived the Examiner about the public distribution of TransWeb's filtration material at the Expo and elsewhere.

At trial, Hanson struggled to explain his conduct. He testified that the reason for his delay in disclosing the reference, until November 2000, was to allow the Confidentiality Agreement between TransWeb and Racal—which he had terminated on April 28, 1999—to expire. (11/19/2012 P.M. Tr. at 58:18-59:23, 60:12-61:1.) But the very MPEP provision that Hanson

cited to the PTO (when requesting expungement of his supplemental disclosure from the public record) states that a patentee must submit material information to the PTO even if it is confidential. Manual of Patent Examining Procedure § 724 (7th ed. July 1998). Hanson knew that the expungement provisions provide a method to protect truly confidential information, so this was not a credible explanation of the delay strategy. Hanson wilted on the witness stand, head down, and, finally, hostile. In the disclosure he finally gave to the Examiner, Hanson stated that "[a]pplicants do believe . . . that the product may have been subsequently commercialized by Transweb" after 3M's patent was filed. (PTX-29.2.) The disclosure also said that "neither the Transweb Tmelt 30P product, nor the product sheet, was shown or otherwise known to the inventors named in this patent application, or the undersigned, before the July 2, 1998 filing date of this [3M] patent application." (*Id.*)

Based on the evidence in this case, these statements were untrue. The only reasonable inference is that Hanson and Jones knew they were not true. When they rushed their own patent through the process, fudging the "witnessing" of the notebooks, they knew that 3M had been focused on TransWeb in advance of the Expo; they knew that TransWeb was at the Expo, distributing its prior art product; they knew that Sundet and Christensen had collected samples; Jones had tested the Racal samples cut from the same roll distributed at the Expo; Legare had PTX-1338 in his files, which was identical to the samples distributed by Ogale at the Expo; and when they were concerned that TransWeb's samples were in fact prior art, they ordered—and Jones tested—examples of the prior art as advertised in *Nonwovens Industry*, confirming that the samples were prior art. They did not disclose any of this information to the Examiner. No mention was made of the T-Melt 50P product, nor of the Willson P-95 filter material, nor of the

PTX-1338 sample pack from the Expo that was in Legare's files.[26] Regrettably, the only reasonable inference that explains the actions of Hanson and Jones is that they delayed in saying anything to the Examiner, and then finally made a materially misleading disclosure.

When the Examiner rejected the patent after later being told about only a minor portion of the prior art (the Racal samples, but not the public distribution at the Expo), Hanson and Jones then enlisted the assistance of outside counsel to further mislead by insisting that all of the prior art was covered by a confidentiality agreement, when it was not.[27] Seen in its best light, Hanson and Jones had a weak argument that one set of samples sent to Legare without a confidentiality agreement were "covered" by the agreement signed a few weeks later. If this were the only prior art, then inequitable conduct would be a much closer call. However, the evidence in this case is powerful that there were multiple sources of prior art known to Hanson, Jones, Rousseau, and others at 3M, and that no confidentiality agreement took that art out of the "public" realm. This entire category of prior art—products from the Expo (as advertised in *Nonwovens Industry*), products sold to Willson—was knowingly omitted from the patent application materials and Supplemental Disclosure because, if the Examiner knew about it, the patent would be rejected without any possible rebuttal argument about confidentiality.

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[26] Hanson did not disclose the T-Melt 50P sample from Racal at all, and had no explanation as to why. (11/20/2012 A.M. Tr. at 6.90:2-91:7.) Importantly, that was the exact product that was in public use in the Willson P-95 respirator, which Hanson and Jones also failed to disclose to the PTO. (11/19/2012 Tr. at 5A.47:9-18; 11/19/2012 P.M. Tr. at 75:17-76:12.) Instead, misleadingly, Hanson's Supplemental IDS stated that the inventors "believe[d] . . . that the product may have been subsequently commercialized by Transweb" after 3M's patent was filed. (PTX-29.2.) In fact, they knew it had been earlier commercialized.

[27] This Court does not find that outside counsel retained for this purpose had knowledge of the falsities perpetrated by Jones and Hanson.

The Court has reviewed the record in exhaustive detail. Inequitable conduct turns on the actions of individuals who owe a duty of candor to the PTO: whether those individuals knew of the prior art reference, knew it was material, knew that it should be disclosed, and intentionally failed to disclose it or misled the PTO about the reference. *Therasense*, 649 F.3d at 1290. The Court finds that the evidence of the inequitable conduct by Hanson and Jones is clear and convincing. *See Avid Identification Sys., Inc. v. Crystal Import Corp.*, 603 F.3d 967, 974 n.1 (Fed. Cir. 2010) ("[O]nly individuals, rather than corporations, owe a duty of candor to the PTO," so "only individuals can breach that duty and give rise to a finding of inequitable conduct."). In this case, the actions of other 3M employees—Rousseau,[28] Legare, Nagel, Lyons, and others—provide strong corroborating evidence of the inequitable conduct of Jones and Hanson. The Court, after receiving full post-trial briefing; conducting its own de novo review of all the facts in the case; after having observed the demeanor of the witnesses; and conducting its own credibility determinations based on those observations, finds that the '458 and '551 patents were the result of inequitable conduct by individuals who owed a duty of candor to the PTO. U.S. Patents 6,397,458 and 6,808,551 are unenforceable. The advisory jury unanimously reached the same conclusion.

[28] In particular, while Rousseau's actions constitute some evidence of the knowledge and intent of Jones and Hanson, TransWeb has not sought to prove by clear and convincing evidence that Rousseau committed inequitable conduct. Rather, his participation in the group actions is included here because he contributed to the conduct of Hanson and Jones. As a named inventor, Rousseau clearly had a duty of candor to the Patent Office, but the extent to which he engaged in inequitable conduct has not been developed or proven factually in this case.

V. JUDGMENT AS A MATTER OF LAW ON ANTITRUST COUNTS

3M also moves for Judgment as a Matter of Law on the antitrust claims. Because the jury returned a verdict in 3M's favor on the sham litigation claim, the motion is moot as to that issue. The Court will therefore address the Rule 50(b) arguments that pertain to *Walker Process* fraud.

As an initial matter, based on the fact findings set forth above concerning inequitable conduct, the Court finds that there was sufficient evidence from which a reasonable jury could conclude that 3M's employees engaged in *Walker Process* fraud by intentionally misleading the Examiner about a material issue.

3M raises the following remaining arguments in challenging the *Walker Process* fraud verdict: A) alleged failure to present evidence of the relevant geographic market, the United States; B) alleged failure to present evidence of an upstream market limited to fluorinated polymeric material for respirators; C) alleged failure to prove that 3M's share of the relevant market is sufficient to constitute a monopoly or a dangerous probability of obtaining a monopoly; D) alleged failure to prove a dangerous probability of obtaining a monopoly and failure to prove anti-competitive effects from the lawsuit; and E) alleged failure to prove antitrust injury to TransWeb.

A. Evidence of the Relevant Geographic Market

"The relevant geographic market is the area in which a potential buyer may rationally look for the goods or services he or she seeks." *Tunis Bros. Co., Inc. v. Ford Motor Co.*, 952 F.2d 715, 726 (3d Cir. 1991) (quoting *United States v. Grinnell Corp.*, 384 U.S. 563, 570-71 (1966)); *Tampa Elec. Co. v. Nashville Coal Co.*, 365 U.S. 320, 327, 331-32 (1961) (defining the relevant geographic area as "the market area in which the seller operates, and to which the purchaser can practicably turn for supplies" or as the area in which suppliers "effectively compete"). "Consequently, the geographic market is not comprised of the region in which the

seller attempts to sell its product, but rather is comprised of the area where his customers would look to buy such a product." *Tunis Bros.*, 952 F.2d at 726.; *see also U.S. Horticultural Supply v. The Scotts Co.*, 367 F. App'x 305, 311 (3d Cir. 2010) (noting that the geographic market is based on buyer behavior). TransWeb bore the burden of proving its relevant geographic market. *Tunis Bros.*, 952 F.2d at 722. On a motion for judgment as a matter of law, the Court must determine whether there were facts from which a reasonable jury could determine that TransWeb had carried its burden. *Cf. Lithuanian Commerce Corp., Ltd. v. Sara Lee Hosiery*, 219 F. Supp. 2d 600, 604 (D.N.J. 2002) (the court grants judgment as a matter of law if "after viewing the evidence in the light most favorable to the nonmovant and giving it the advantage of every fair and reasonable inference, there is insufficient evidence from which a jury reasonably could find liability.").

Here, there was evidence from which a jury could conclude that the geographic market was the United States for the downstream market in NIOSH-certified respirators. First, NIOSH is a U.S. agency that sets standards for respirators used by companies operating within the U.S. so that they may provide their U.S.-based workers with respiratory protection that complies with the U.S. Occupational Safety & Health Administration's regulations. (11/14/2012 A.M. Tr. at 2A.77:15-21; 11/15/2012 P.M. Tr. at 4.11:9-18; 11/16/2012 Tr. at 4.28:9-29:6). Second, 3M's own internal documents divide geographic markets into the U.S. and other regions, and assess competition from such respirator manufacturers within the U.S. (PTX-125.4; PTX-132.3, 6-8, 12; PTX-245.2-3; DTX-316 (detailing U.S. sales)). Third, TransWeb's economics expert, Dr. Bradley Reiff—the only antitrust expert in this case—testified that the geographic market was the U.S., which was based on his previously-uncontested expert opinion. (11/26/2012 P.M. Tr. at 14:3-10, 29:2-20.)

Similarly, evidence was introduced that the geographic market for the upstream market in filtration material was the United States. It was undisputed that the only merchant supplier—*i.e.*, company selling fluorinated polymeric media—in the world is TransWeb. (11/26/2012 A.M. Tr. at 7A.15:7-15:15). Thus, the only place "in which a potential buyer may rationally look for the goods or services he seeks"—in this case, plasma fluorinated polymeric filtration media—is the U.S. *See Gordon v. Lewistown Hosp.*, 423 F.3d 184, 212 (3d Cir. 2005).

B. Evidence of an Upstream Market Limited to Fluorinated Polymeric Material for Respirators

The relevant product market must include all products that are "roughly equivalent to [one] another for the use to which [they are] put" even if there "may be some degree of preference for the one over the other." *Queen City Pizza, Inc. v. Domino's Pizza, Inc.*, 124 F.3d 430, 436-37 (3d Cir. 1997); *Town Sound & Custom Tops, Inc. v. Chrysler Motors Corp.*, 959 F.2d 468, 480 (3d Cir. 1992) (en banc) (finding that the market includes "Chrysler cars and cars that are reasonably interchangeable with Chrysler cars"); *Crestron Elecs., Inc. v. Cyber Sound & Sec. Inc.*, Civ. No. 11-3492, 2012 WL 426282, at *6 (D.N.J. Feb. 9, 2012) ("[T]he interchangeability of these components produced by different manufacturers is vital to defining the relevant product market.").[29] "The product market is defined by reference to the reasonable

[29] A brand name alone does not indicate a distinct submarket—Domino's competes with other pizza delivery companies, Chrysler with other car manufacturers, and Crestron with other home electronic system developers. A plaintiff must point to additional indicia of the submarket, such as distinctive physical characteristics or uses, in addition to price differences and consumer preferences. Those indicia must be tested for reasonable interchangeability, as Reiff did here. Definition of the relevant market is a question of fact. *See, e.g.*, *IGT v. Alliance Gaming Corp.*, 702 F.3d 1338, 1344 (Fed. Cir. 2012). In this case, there was sufficient evidence that both the upstream and downstream markets are distinctive—physically, chemically, in their price and performance, and in the minds of consumers—for the question to have gone to the jury. 3M's arguments now go to weight, but that is not the standard on a motion for judgment as a matter of law. This question has been decided by the jury, and 3M has not made a showing that the Court should set aside the verdict.

interchangeability in use among competing products or by reference to the cross-elasticity of demand between a product and its substitutes." *Delano Farms Co. v. Cal. Table Grape Comm'n*, 655 F.3d 1337, 1351 (Fed. Cir. 2011) (citing, *inter alia*, *Brown Shoe Co. v. United States*, 370 U.S. 294, 325 (1962)). In defining a submarket for antitrust purposes, plaintiffs may rely on "industry or public recognition of the submarket as a separate economic entity, the product's peculiar characteristics and uses, unique production facilities, distinct customers, distinct prices, sensitivity to price change, and specialized vendors." *Brown Shoe*, 370 U.S. at 325.

Here, there was evidence upon which the jury could rely in concluding that TransWeb proved that fluorinated polymeric material for respirators is a distinct market from other types of filtration material. First, evidence was introduced that fluorinated polymeric media has the best combination of low pressure drop and high filtration efficiency of all types of respirator filtration media. (11/26/2012 A.M. Tr. at 7A.50:14-22; PTX-245.4.) Second, fiberglass filters have much higher pressure drop than fluorinated polymeric media because they rely solely on mechanical filtration (as opposed to electret filtration). (11/27/2012 P.M. Tr. at 79:13-24; 11/14/2012 A.M. Tr. at 2A.5:19-20, 8:18-20.) Third, evidence adduced at trial indicated fluorinated polymeric filtration media allows respirators to have a lower pressure drop and longer service life than respirators incorporating other types of filtration media. (11/14/2012 A.M. Tr. at 9:12-10:23; PTX-125.2, 4, 6, 11; PTX-245.4.) Fourth, there was evidence that using fluorinated polymeric media in a respirator lowers the total cost of ownership of such respirators because they last longer, which is a competitive advantage that 3M advertises. (11/26/2012 A.M. Tr. at 7A.26:7-27:13; 11/26/2012 P.M. Tr. at 66:6-67:3; PTX-125.11.) Finally, 3M's Voice of the Customer consumer surveys indicate that consumers will pay more for the lower pressure drop and longer service life that fluorinated polymeric filtration media provides to respirators. (PTX-125.4).

C. 3M's Share of the Relevant Market

"Monopoly power is the ability to control prices and exclude competition in a given market. If a firm can profitably raise prices without causing competing firms to expand output and drive down prices, that firm has monopoly power." *Broadcom Corp. v. Qualcomm Inc.*, 501 F.3d 297, 307 (3d Cir. 2007) (internal citations omitted).

Here, 3M's potential monopoly power over the upstream market in the media (had it excluded TransWeb from the market) cannot be disputed, because TransWeb is the only other supplier of the media. (11/26/2012 A.M. Tr. at 7A.15:7-15:15.)

The jury could have relied on Reiff's testimony in concluding that there was a dangerous probability that 3M would achieve a monopoly in the downstream market in respirators as well. Reiff testified that in reaching his conclusion, he used 3M and TransWeb's actual costs and sales data to determine the relative share of all 3M R and P respirators as compared to all R and P respirators using TransWeb's fluorinated filtration media.[30] (11/26/2012 P.M. Tr. at 35:4-36:3). This market share analysis included both disposable and reusable respirators, and included all non-3M, non-TransWeb media respirators. (*Id.* at 33:12-36:3). Moreover, evidence was introduced that there are barriers to entry in the market. [31,32] Reiff testified that there are barriers

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[30] The sales data on which Reiff relied were introduced into evidence. (*See* PTX-119; 123; 132; 134; 242; 245; 290; 291; 1114; 1115; 1117; 1118; 1119; 1120; 1121; 1306; and 1307.)

[31] *Broadcom*, 501 F.3d at 307 ("Barriers to entry are factors, such as regulatory requirements, high capital costs, or technological obstacles, that prevent new competition from entering a market in response to a monopolist's supracompetitive prices.") Here, NIOSH approval clearly serves as a regulatory barrier, although that alone is insufficient to show potential monopoly power. *See Barr Labs., Inc. v. Abbott Labs.*, 978 F.2d 98, 113 (3d Cir. 1992) (FDA approval alone is not a barrier to entry).

[32] 3M's only evidence in opposition to this conclusion is the testimony of Vaughn Grannis, a 3M employee, that the reusable segment of the market is larger than the disposable segment, and that 3M's share of the disposable segment is larger than its share of the reusable segment.

to entry, as demonstrated by 3M's current high market share and extremely high profit margins on its R and P respirator products. (11/26/2012 P.M. Tr. at 83:18-24).

D. Dangerous Probability of Obtaining a Monopoly; Anti-Competitive Effects

"The offense of attempted monopolization need not cause actual market damage, but need merely threaten to produce the type of market damage contemplated in the antitrust laws." *Multiflex, Inc. v. Samuel Moore & Co.*, 709 F.2d 980, 994 (5th Cir. 1983). 3M argues that TransWeb has not been eliminated, and cannot be eliminated unless 3M won the lawsuit, so there is no dangerous probability. That reasoning is entirely circular and does not correctly state the law. *See, e.g.*, *Nobelpharma AB v. Implant Innovations, Inc.*, 141 F.3d 1059, 1068-69 (Fed. Cir. 1998) (noting *Walker Process* fraud liability attaches when patentee obtained patent by fraud and was aware of that fraud "when bringing suit"); *see also Multiflex*, 709 F.2d at 994 ("The mere attempt [to monopolize] is the offense.").

3M also contends that "[a]ll Section 2 claims—including attempted monopolization—require a showing that competition was harmed by the defendant's conduct." (Mem. in Supp. of 3M's Mot. for J. as a Matter of Law on the Antitrust Claim 14-15, ECF No. 494 (citing *Spectrum Sports, Inc. v. McQuillan*, 506 U.S. 447, 455 (1993); *Race Tires Am., Inc. v. Hoosier Racing Tire Corp.*, 614 F.3d 57, 75 (3d Cir. 2010)).) However, in the attempted monopolization context, the test "is whether the 'dangerous probability of success,' *if it had materialized*, would have caused the type of market damage the antitrust laws seek to prevent." *Multiflex*, 709 F.2d at 994 (emphasis in original).

(11/26/2012 Tr. at 7A.79:1-5.) Reiff testified that Grannis's claim includes N respirators, which may skew the market share estimates in the reusable segment, and that he had seen no documentary evidence to support that claim. (11/26/2012 P.M. Tr. at 72:25-73:20, 75:11-76:12.) This is an issue of fact that the jury decided, based on the competing evidence in the case. There was sufficient evidence upon which the jury rested its verdict.

E. Antitrust Injury

3M argues that attorneys' fees cannot be antitrust damages because TransWeb must show that paying the fees caused the disruption in a relevant market. This misstates the law in several ways. First, a *Walker Process* fraud claim where a defendant attempts to monopolize does not require actual disruption—it requires a showing that the fraud "threaten[s] to produce the type of market damage contemplated in the antitrust laws." *Multiflex*, 709 F.2d at 994. Second, the antitrust damages need not be identical to the antitrust injury—they only need to flow from the anticompetitive conduct. *See, e.g.*, *Rossi v. Standard Roofing, Inc.*, 156 F.3d 452, 483 (3d Cir. 1998) (the proof for antitrust damages is whether "*some* damage flow[s]" from the anticompetitive conduct) (emphasis in original). Attorneys' fees can be the damages that flow from the injury. *Handgards, Inc. v. Ethicon, Inc.*, 601 F.2d 986, 997 (9th Cir. 1979) ("In a suit alleging antitrust injury based upon a bad faith prosecution theory it is obvious that the costs incurred in defense of the prior patent infringement suit are an injury which 'flows' from the antitrust wrong."); *Kearney & Trecker Corp. v. Cincinnati Milacron, Inc.*, 562 F.2d 365, 374 (6th Cir. 1977) ("When the antitrust violations are causally connected to the infringement action it is permissible to include the expenses of defending that action in the award of damages."); *see also Bristol-Myers Squibb Co. v. Ben Venue Labs.*, 90 F. Supp. 2d 540, 543-45 (D.N.J. 2000) (relying on *Handgards* to find that an accused infringer claiming *Walker Process* fraud in an ANDA case can rely on the costs of suit to show injuries).

TransWeb adduced sufficient evidence of its antitrust claims to support the jury's verdict. 3M's motion is denied.[33]

[33] 3M also moved for judgment as a matter of law that TransWeb willfully infringed the 3M '458 patent claims. In light of the jury's invalidity verdict and the Court's decision on unenforceability, the Court need not address the motion, and it is therefore terminated as moot.

VI. ATTORNEYS' FEES

3M challenges the calculation of TransWeb's award of attorneys' fees. By consent of the parties, this Court referred the determination of attorneys' fees, in light of the jury verdict for TransWeb, to a Special Master, Hon. Alfred M. Wolin [ECF No. 523]. Judge Wolin was tasked with calculating both the antitrust cost of suit and antitrust damages. The cost of suit included the attorneys' fees TransWeb paid to its attorneys from the firm Quinn Emanuel Urquhart & Sullivan, LLP, to successfully prosecute its antitrust claims against 3M. Because the jury found that TransWeb suffered antitrust injury—in the form of paying patent defense attorneys' fees—antitrust damages include the attorneys' fees TransWeb paid to defend against 3M's patent suit. As all parties agreed, fees related to patent defense shall be trebled as antitrust damages, whereas fees related to TransWeb's antitrust claims are recoverable on a one-to-one basis as costs of suit.

The parties spent months presenting their evidence and arguments to Judge Wolin. The Special Master issued a Report and Recommendation awarding a total of $26,146,493.45 in attorneys' fees and costs [ECF No. 567]. Pursuant to Federal Rule of Civil Procedure 53, Defendants filed objections to the Report & Recommendation and seek de novo review of the findings. They argue that the Special Master's calculations are erroneous: because TransWeb's billing entries are purportedly too vague to distinguish between patent defense fees (subject to trebling) and antitrust prosecution fees (not trebled); and because the attorneys' fees TransWeb paid to the law firm Quinn Emanuel to litigate this case are allegedly above the market rate.

As the parties agree, TransWeb's burden to establish the amount of antitrust cost of suit—antitrust litigation fees—may be satisfied by a lodestar analysis, whereas TransWeb's burden to prove the amount of antitrust damages—patent litigation fees—is satisfied by providing a "reasonable estimate." (Report & Recommendation 10, ECF No. 567). In

determining whether TransWeb had proffered a reasonable estimate of antitrust damages, the Special Master undertook an exhaustive analysis of the billing entries and timekeeping procedures by TransWeb's attorneys. Although some of Quinn Emanuel's billing entries were labeled as either "antitrust" or "patent," other block-billed entries represented hours spent on mixed patent defense and antitrust litigation issues, e.g., "prepared for trial." TransWeb consented to treating any block-billed entry that contained even a single explicit reference to the antitrust claims entirely as antitrust fees to eliminate the possibility of improper trebling. Judge Wolin and the parties examined the remaining block-billed entries to determine which portion should be designated as patent defense fees, subject to trebling, and which portion should be designated as antitrust litigation fees.

Judge Wolin's well-considered analysis reflects months of examining hundreds of time entries billed by Quinn Emmanuel, affidavits regarding reasonable billing rates, and declarations attesting to the basis for designation of the block-billed fees. Based on that evidence, and confirmed by the history of this litigation, Judge Wolin found that this case was primarily about patent defense. Consequently, for block-billed entries that did not specify whether the work was related to patent or antitrust issues, the Special Master assigned a presumptive weight of 75% patent and 25% antitrust fees. On June 5, 2013, Judge Wolin held a ten-hour in-person hearing and heard argument on the allocation of hours for the 656 challenged block-billed entries, as related either to patent defense or antitrust litigation, on an entry-by-entry basis. The Special Master also discussed the reasonableness of Quinn Emanuel's hourly rate and whether the amount of time spent on each challenged task was reasonably expended. Based on this hearing, and the context of the surrounding entries, Judge Wolin adjusted the presumptive percentage for some of the block-billed entries. He also discounted some entries after determining that the work,

though done by an attorney, could have been performed by a paralegal. He noted that TransWeb had voluntarily adopted a conservative approach to billing, did not seek reimbursement of miscellaneous billing items, and did not request fees for work related to local counsel, paralegals, or non-attorney support staff.

Defendants contend that the Special Master erred by allocating approximately 75% of the challenged block-billing entries as patent defense work, subject to trebling. 3M argues that, for any block-billed entry that includes mixed antitrust and patent issues, the entire entry should be treated as antitrust litigation fees and recovered on a one-to-one basis. They request a corresponding reduction in fees of $1,405,935.54 from the $26,146,493.45 total. The Court disagrees. Judge Wolin's detailed analysis of the nature of the case and the challenged entries supports the proportion allocated to antitrust fees and represents a reasonable estimate of antitrust damages.

Defendants further argue that the hourly rate at which Quinn Emanuel billed its client should be discounted by twenty percent, purportedly to reflect the prevailing rates at which attorneys charge for patent litigation in New Jersey. Examining whether the rates that Quinn Emanuel charged were a reasonable market rate based on the complexity of the services, the Special Master considered the affidavits of other attorneys from national firms and compared the rate to attorneys of similar skill and experience. Accordingly, he found that TransWeb's choice of Quinn Emanuel, although not a New Jersey firm, was reasonable given that the survival of the company required successfully defending a patent case against a multinational corporation with extensive resources.[34] As Judge Wolin noted, 3M is currently engaged in 31 patent cases and has

[34] Evidence adduced at trial showed that, when Ogale was sued by 3M, he was forced by economic realities of the cost of defense to sell a major stake in his small company to an investor

the "economic muscle" to demand discounts from top law firms. TransWeb, a firm that had 55 employees at its peak, does not have similar bargaining power. The Court agrees with the Special Master that, because TransWeb's existence hinged upon winning a complex patent litigation with novel and difficult substantive issues, a premier litigation firm was a reasonable choice.

VII. CONCLUSION

For the foregoing reasons, 3M's motions for judgment as a matter of law as to TransWeb's antitrust claim and its invalidity claims is denied, and the Court finds that 3M's patents, '458 and '551, are unenforceable due to inequitable conduct. The Special Master's calculation of attorneys' fees is found to be properly analyzed and correctly calculated. An appropriate Order will follow.

IT IS SO ORDERED

/s Faith S. Hochberg
Hon. Faith S. Hochberg, U.S.D.J.

with resources to finance the litigation defense costs. He could not afford to keep his company's ownership for himself, even if he were able to win the litigation. (11/14/2012 P.M. Tr. at 82:1-15.)

TAB 2

NOT FOR PUBLICATION

UNITED STATES DISTRICT COURT
DISTRICT OF NEW JERSEY

TRANSWEB, LLC, Plaintiff, v. 3M INNOVATIVE PROPERTIES COMPANY and 3M COMPANY, Defendants.	Hon. Faith S. Hochberg, U.S.D.J. Civil Case No. 10-4413 (FSH) (JBC) **ORDER** Date: April 21, 2014

HOCHBERG, District Judge:

For the reasons set forth in the Court's Opinion of April 21, 2014;

IT IS, on this 21st day of April, 2014, hereby

ORDERED that Defendants' Motions for Judgment as a Matter of Law on Invalidity [Docket No. 492]; and Judgment as a Matter of Law on the Antitrust Claim [Docket No. 494] are **DENIED**;

IT IS FURTHER ORDERED that judgment shall be entered in accordance with the jury's finding that claims 57 and 31 of United States Patent Nos. 6,397,458 are invalid as obvious;

IT IS FURTHER ORDERED that Defendants' Motion for Judgment as a Matter of Law of Law on Infringement and Willfulness [Docket No. 497] is **DENIED** as moot;

IT IS FURTHER ORDERED that, based on the Court's findings, the Court holds U.S. Patent Nos. 6,397,458 and 6,808,551 unenforceable as a result of inequitable conduct;

IT IS FURTHER ORDERED that the Court adopts the Report and Recommendation of the Special Master [Docket No. 567] regarding attorneys' fees and costs;

IT IS FURTHER ORDERED that the parties shall submit a form proposed judgment within seven (7) days.

IT IS SO ORDERED

/s Faith S. Hochberg
Hon. Faith S. Hochberg, U.S.D.J.

TAB 3

CLOSED

UNITED STATES DISTRICT COURT
FOR THE DISTRICT OF NEW JERSEY

TRANSWEB, LLC, Plaintiff, v. 3M INNOVATIVE PROPERTIES COMPANY, ET AL., Defendants.	Civil Action No. 10-4413(FSH)(JBC) Hon. Faith S. Hochberg, U.S.D.J. ~~[PROPOSED]~~ **FINAL JUDGMENT**

WHEREAS, TransWeb, LLC ("TransWeb") filed this action on August 27, 2010, against 3M Company and 3M Innovative Properties Company (collectively, "3M") seeking a declaratory judgment of invalidity of claims in U.S. Patent Nos. 6,397,458 ("the '458 patent") and 6,808,551 ("the '551 patent") of which 3M is the assignee, unenforceability of those patents due to 3M's inequitable conduct in the prosecution of those patents, and non-infringement by TransWeb of those patents; and TransWeb subsequently filed an amended complaint stating, *inter alia*, antitrust claims of *Walker Process* fraud and sham litigation;

WHEREAS, 3M counterclaimed for patent infringement;

WHEREAS, 3M voluntarily dismissed with prejudice before trial its claims under the '551 patent;

WHEREAS, trial commenced on November 13, 2012 on TransWeb's antitrust and patent declaratory judgment claims and 3M's remaining patent infringement counterclaims;

WHEREAS, at the close of TransWeb's case, 3M moved for judgment as a matter of law pursuant to Federal Rule of Civil Procedure 50 on the claims of invalidity, infringement and willfulness, inequitable conduct, and antitrust claims, and the Court reserved on those motions;

WHEREAS, at the conclusion of the trial, the jury returned a verdict finding that the 3M patent claims asserted at trial were invalid as obvious, that TransWeb did not infringe 3M's patent claims asserted at trial, and that 3M violated the antitrust laws by enforcing or attempting to enforce fraudulently procured patents but that 3M did not engage in sham litigation in violation of the antitrust laws; the jury returned a unanimous advisory verdict that the '458 and '551 patents were unenforceable due to inequitable conduct and the jury found that TransWeb was entitled to $34,412 in lost profits and an undetermined amount of attorneys' fees based on 3M's violation of the antitrust laws;

WHEREAS, 3M then renewed its motions for judgment as a matter of law, and the Court instructed the parties to brief those issues as well as whether the Court should find the patents unenforceable due to inequitable conduct; and the Court held a post-trial hearing on January 10, 2013; and

WHEREAS, by consent of the parties, this Court referred the determination of the amount of TransWeb's attorneys' fees to a Special Master, Hon. Alfred M. Wolin (Ret.); the Special Master was tasked with calculating both the patent defense fees which were recoverable as antitrust damages, as well as reasonable attorneys' fees as antitrust costs of suit under the Clayton Act; the Special Master submitted a Report and Recommendation to this Court on September 24, 2013 [Docket No. 567], to which 3M filed objections and TransWeb filed a response;

WHEREAS, on April 21, 2014 this Court issued an Opinion [Docket No. 579] and Order [Docket No. 580] denying 3M's Motions for Judgment as a Matter of Law on Invalidity [Docket No. 492] and Judgment as a Matter of Law on the Antitrust Claim [Docket No. 494], and denying as moot 3M's Motion for Judgment as a Matter of Law of Law on Infringement and

Willfulness [Docket No. 497]; further finding that the '458 and '551 patents are unenforceable due to inequitable conduct; and adopting the Special Master's Report and Recommendation on attorneys' fees and costs in its entirety;

NOW THEREFORE, IT IS HEREBY ORDERED, ADJUDGED, AND DECREED as follows:

1. Final Judgment is entered in favor of Plaintiff TransWeb and against Defendants 3M Company and 3M Innovative Properties Company (collectively "3M") as follows:

   a. Judgment is entered, in accordance with the jury's finding, that claims 31 and 57 of the '458 patent are invalid as obvious;

   b. Judgment is entered, in accordance with the jury's finding, that claims 31 and 57 of the '458 patent were not infringed by TransWeb;

   c. Judgment is entered, in accordance with the jury's findings, that 3M shall take nothing from its counterclaims against TransWeb;

   d. Judgment is entered, in accordance with the jury's finding, that 3M violated the antitrust laws by enforcing or attempting to enforce fraudulently procured patents against TransWeb;

   e. Antitrust damages are awarded to TransWeb and against 3M in the amount of $23,074,306.48, consisting of: (1) $34,412.00 in lost profits, trebled to $103,236.00; and (2) $7,657,023.49 in patent defense fees, trebled to $22,971,070.48;[1]

[1] This amount is calculated by analyzing the exhibits to the Special Master's Report and Recommendations [Docket No. 567, 568]. Exhibit A, reflecting the billing entries for which 3M did not challenge the designations, reflects a recommended award of $18,400,816.50 (after

f. Reasonable attorneys' fees as antitrust costs of suit are awarded to TransWeb and against 3M in the amount of $3,175,422.97, which amount is not trebled;[2]

g. Judgment is entered, in accordance with the Court's findings and the jury's unanimous advisory verdict, that the '458 patent and the '551 patent are unenforceable as a result of inequitable conduct by Karl Hanson and Marvin Jones;

2. Final judgment shall be entered in favor of 3M and against TransWeb on TransWeb's claim that 3M violated the antitrust laws by engaging in sham litigation.

3. TransWeb may seek its costs of suit in accordance with the provisions of L. Civ. R. 54.1.

4. TransWeb is entitled to post-judgment interest on this Judgment consistent with applicable law.

SO ORDERED this 29 day of April 2014.

HON. FAITH S. HOCHBERG, U.S.D.J.

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trebling) in patent defense fees and $2,372,735.50 in antitrust "costs of suit." *See* Docket No. 567 (Report & Recommendations) at 40 & Docket No. 568 (Ex. A). Exhibit B, reflecting the billing entries for which the parties reached agreement during the hearing before the Special Master, reflects a recommended award of $58,758.00 (after trebling) in patent defense fees and $401,816.75 in antitrust "costs of suit." *See* Docket No. 567 at 40-41 & Docket No. 568 (Ex. B). Exhibit C, reflecting the billing entries disputed by the parties, reflects a recommended award of $4,511,495.98 (after trebling) in patent defense fees and $400,870.72 in antitrust "costs of suit." *See* Docket No. 567 at 41-43 & Docket No. 568 (Ex. C.).

[2] *See supra* n.1.

TAB 4

NOT FOR PUBLICATION

UNITED STATES DISTRICT COURT
DISTRICT OF NEW JERSEY

TRANSWEB, LLC, Plaintiff, v. 3M INNOVATIVE PROPERTIES COMPANY, et al., Defendants.	Civil Case No. 10-4413 (FSH) **ORDER** June 11, 2014

HOCHBERG, District Judge:

This matter comes before the Court upon Defendants' motion for a new trial [Dkt. No. 589]. Defendants assert, generally, that the jury's verdict would effect a miscarriage of justice. For the reasons set forth at length in this Court's Opinion and Order [Dkt. Nos. 579 & 580], the Court finds that the jury's verdict was correct and amply supported by the evidence. Defendants' motion is denied.

ACCORDINGLY, IT IS, this 11th day of June, 2014, hereby

ORDERED that Defendants' Motion for a New Trial [Dkt. No. 589] is **DENIED**.

IT IS SO ORDERED

/s/ Faith S. Hochberg
Hon. Faith S. Hochberg, U.S.D.J.

TAB 5

VERDICT FORM:
TransWeb, LLC v. 3M Innovative Properties Co., *et al.*

UNITED STATES DISTRICT COURT
DISTRICT OF NEW JERSEY

TRANSWEB, LLC,

Plaintiff,

v.

3M INNOVATIVE PROPERTIES COMPANY and 3M COMPANY,

Defendants.

Civil Action No. 2:10-cv-04413 (FSH/PS)

JURY VERDICT FORM

VERDICT FORM:
TransWeb, LLC v. 3M Innovative Properties Co., *et al.*

We, the jury in the above-titled action, hereby find as follows:

1. Is claim 31 of the '458 patent invalid as obvious?

("Yes" is a finding for TransWeb. "No" is a finding for 3M.)

YES ✓ NO _____

2. Is claim 57 of the '458 patent invalid as obvious?

("Yes" is a finding for TransWeb. "No" is a finding for 3M.)

YES X NO _____

3. Is the '458 patent unenforceable by virtue of inequitable conduct?

(Your finding on this issue is in an advisory capacity. Nonetheless, you should treat this as seriously as the other questions you are answering. If you are unable to reach a unanimous verdict on this question, please record the total number of Yes and No votes and move on to the next question. If you reach a unanimous verdict on this question, please just mark the corresponding answer below with an "X.")

("Yes" is a finding for TransWeb. "No" is a finding for 3M.)

YES X NO _____

4. Is the '551 patent unenforceable by virtue of inequitable conduct?

(Your finding on this issue is in an advisory capacity. Nonetheless, you should treat this as seriously as the other questions you are answering. If you are unable to reach a unanimous verdict on this question, please record the total number of Yes and No votes and move on to the next question. If you reach a unanimous verdict on this question, please just mark the corresponding answer below with an "X.")

("Yes" is a finding for TransWeb. "No" is a finding for 3M.)

YES X NO _____

VERDICT FORM:
TransWeb, LLC v. 3M Innovative Properties Co., *et al.*

5. Did 3M violate the antitrust laws by enforcing or attempting to enforce fraudulently-procured patents?

("Yes" is a finding for TransWeb. "No" is a finding for 3M.)

YES X NO ____

6. Did 3M violate the antitrust laws by engaging in sham litigation?

("Yes" is a finding for TransWeb. "No" is a finding for 3M.)

YES ____ NO X

7. If 3M violated the antitrust laws, is TransWeb entitled to its attorneys' fees?

("Yes" is a finding for TransWeb. "No" is a finding for 3M.)

YES X NO ____

8 If 3M violated the antitrust laws, is TransWeb entitled to lost profits in the amount of $34,412.00?

("Yes" is a finding for TransWeb. "No" is a finding for 3M.)

YES X NO ____

9. Is claim 31 of the '458 patent infringed by TransWeb?

("Yes" is a finding for 3M. "No" is a finding for TransWeb.)

YES ____ NO X

10. Is claim 57 of the '458 patent infringed by TransWeb?

("Yes" is a finding for 3M. "No" is a finding for TransWeb.)

YES ____ NO X

VERDICT FORM:
TransWeb, LLC v. 3M Innovative Properties Co., *et al.*

Your deliberations are at an end and you should review the verdict form and ensure it accurately reflects your unanimous determinations. The jury foreperson should then sign and date this Verdict Form and notify the bailiff that you have reached a verdict.

Date: 11/30, 2012

TAB 6

US006397458B1

(12) **United States Patent**
Jones et al.

(10) **Patent No.: US 6,397,458 B1**
(45) **Date of Patent: Jun. 4, 2002**

(54) **METHOD OF MAKING AN ELECTRET ARTICLE BY TRANSFERRING FLUORINE TO THE ARTICLE FROM A GASEOUS PHASE**

(75) Inventors: **Marvin E. Jones**, Grant Township; **Christopher S. Lyons**; **David B. Redmond**, both of St. Paul; **Jeffrey L. Solomon**, Vadnais Heights; **Seyed Abolhassan Angadjivand**, Woodbury, all of MN (US)

(73) Assignee: **3M Innovative Properties Company**, St. Paul, MN (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: **09/478,658**

(22) Filed: **Jan. 6, 2000**

Related U.S. Application Data

(62) Division of application No. 09/109,497, filed on Jul. 2, 1998.

(51) **Int. Cl.**[7] **H01R 43/00**
(52) **U.S. Cl.** **29/825**; 29/592.1; 29/826
(58) **Field of Search** 29/825, 826, 592.1, 29/830, 832, 833

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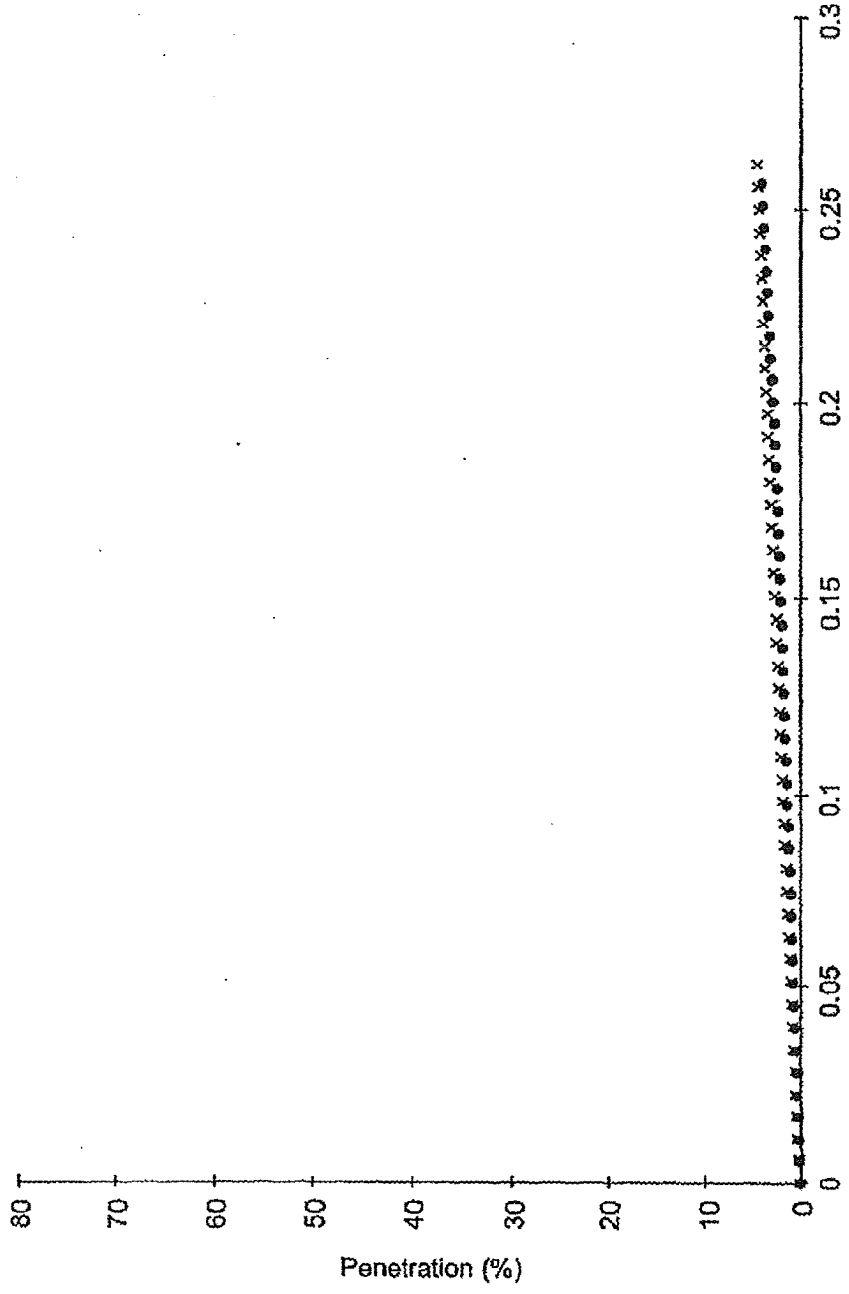
Primary Examiner—Carl J. Arbes
(74) *Attorney, Agent, or Firm*—Karl G. Hanson; Allison Johnson

(57) **ABSTRACT**

An electret is described that includes a surface modified polymeric article having surface fluorination produced by fluorinating the polymeric article.

58 Claims, 3 Drawing Sheets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0.25
0.2
0.15
0.1
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70
60
50
40
30
20
10
0
Penetration (%)

Fig. 1

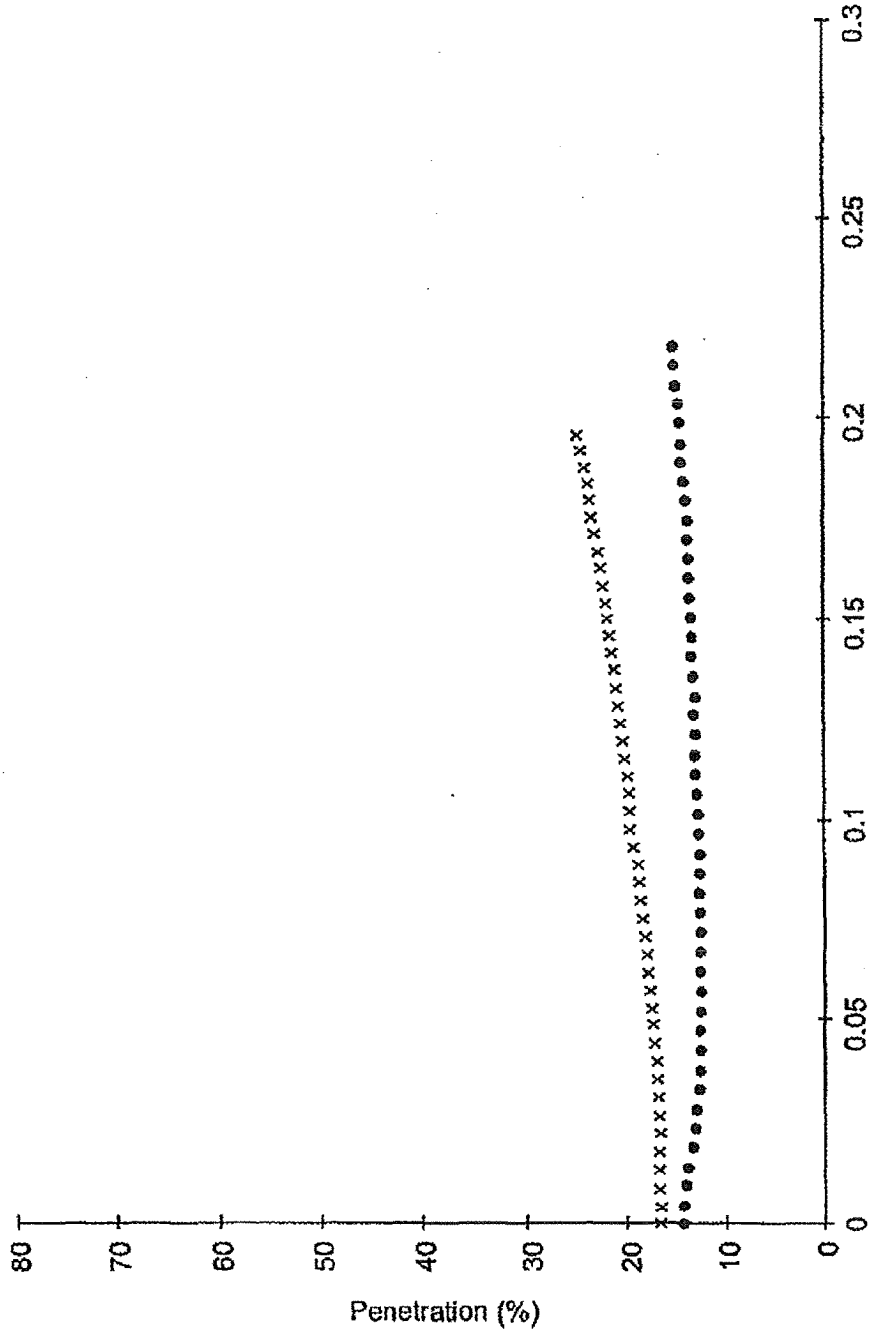

DOP Load (grams)
0
0.05
0.1
0.15
0.2
0.25
0.3
Penetration (%)
80
70
60
50
40
30
20
10
0

Fig. 2

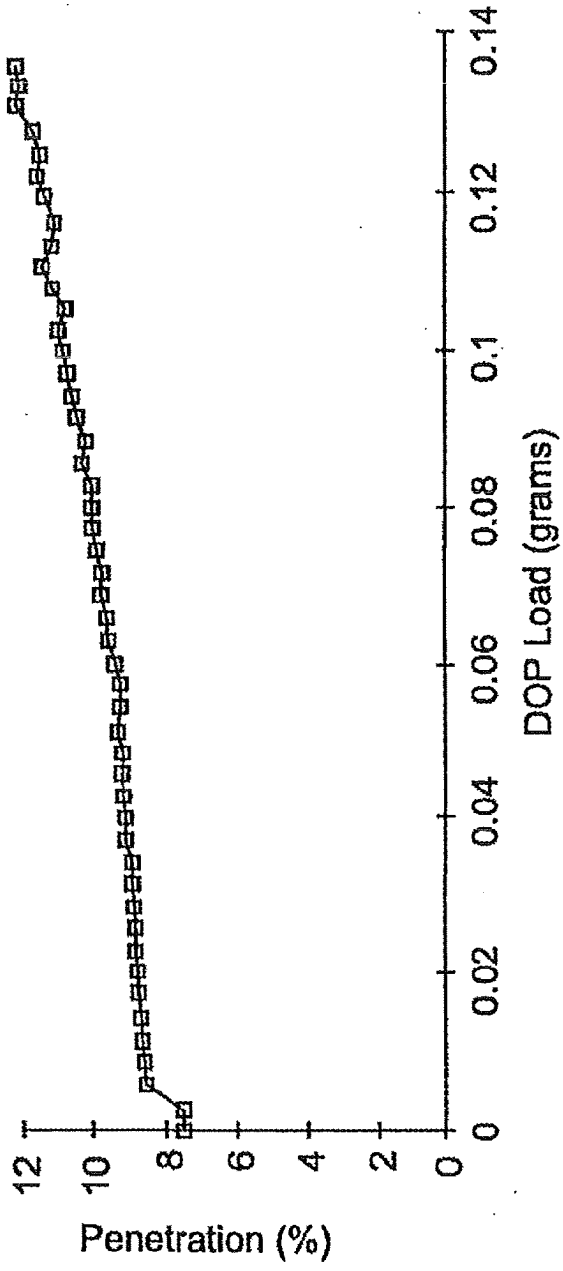

0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
DOP Load (grams)
12
10
8
6
4
2
0
Penetration (%)

Fig. 3

METHOD OF MAKING AN ELECTRET ARTICLE BY TRANSFERRING FLUORINE TO THE ARTICLE FROM A GASEOUS PHASE

This is a divisional of application Ser. No. 09/109,497 filed Jul. 2, 1998.

This invention relates to preparing fluorinated electrets.

BACKGROUND

The filtration properties of nonwoven polymeric fibrous webs can be improved by transforming the web into an electret, i.e., a dielectric material exhibiting a quasi-permanent electrical charge. Electrets are effective in enhancing particle capture in aerosol filters. Electrets are useful in a variety of devices including, e.g., air filters, face masks, and respirators, and as electrostatic elements in electro-acoustic devices such as microphones, headphones, and electrostatic recorders.

Electrets are currently produced by a variety of methods including direct current ("DC") corona charging (see, e.g., U.S. Pat. No. 30,782 (van Turnhout)), and hydrocharging (see, e.g., U.S. Pat. No. 5,496,507 (Angadjivand et al.)), and can be improved by incorporating fluorochemicals into the melt used to produce the fibers of some electrets (see, e.g., U.S. Pat. No. 5,025,052 (Crater et al.)).

Many of the particles and contaminants with which electret filters come into contact interfere with the filtering capabilities of the webs. Liquid aerosols, for example, particularly oily aerosols, tend to cause electret filters to lose their electret enhanced filtering efficiency (see, e.g., U.S. Pat. No. 5,411,576 (Jones et al.)).

Numerous methods have been developed to compensate for loss of filtering efficiency. One method includes increasing the amount of the nonwoven polymeric web in the electret filter by adding layers of web or increasing the thickness of the electret filter. The additional web, however, increases the breathing resistance of the electret filter, adds weight and bulk to the electret filter, and increases the cost of the electret filter. Another method for improving an electret filter's resistance to oily aerosols includes forming the electret filter from resins that include melt processable fluorochemical additives such as fluorochemical oxazolidinones, fluorochemical piperazines, and perfluorinated alkanes. (See, e.g., U.S. Pat. No. 5,025,052 (Crater et al.)). The fluorochemicals should be melt processable, i.e., suffer substantially no degradation under the melt processing conditions used to form the microfibers that are used in the fibrous webs of some electrets. (See, e.g., WO 97/07272 (Minnesota Mining and Manufacturing)).

SUMMARY OF THE INVENTION

In one aspect, the invention features an electret that includes a surface modified polymeric article having surface fluorination produced by fluorinating a polymeric article. In one embodiment, the article includes at least about 45 atomic % fluorine as detected by ESCA. In another embodiment, the article includes a CF_3:CF_2 ratio of at least about 0.25 as determined according to the Method for Determining CF_3:CF_2. In other embodiments, the article includes a CF_3:CF_2 ratio of at least about 0.45 as determined according to the Method for Determining CF_3:CF_2.

In one embodiment, the article has a Quality Factor of at least about 0.25/mmH_2O, (preferably at least about 0.5/mmH_2O, more preferably at least about 1/mmH_2O).

In some embodiments, the article includes a nonwoven polymeric fibrous web. Examples of suitable fibers for the nonwoven polymeric fibrous web include polycarbonate, polyolefin, polyester, halogenated polyvinyl, polystyrene, and combinations thereof. Particularly useful fibers include polypropylene, poly-(4-methyl-1-pentene), and combinations thereof. In one embodiment, the article includes melt-blown microfibers.

In another aspect, the invention features an electret that includes a polymeric article having at least about 45 atomic % fluorine as detected by ESCA, and a CF_3:CF_2 ratio of at least about 0.45 as determined according to the Method for Determining CF_3:CF_2. In another embodiment, the electret includes at least about 50 atomic % fluorine as detected by ESCA, and a CF_3:CF_2 ratio of at least about 0.25 as determined according to the Method for Determining CF_3:CF_2.

In other aspects, the invention features a respirator that includes the above-described electrets. In still other aspects, the invention features a filter that includes the above-described electrets.

In one aspect, the invention features a method of making an electret that includes: (a) fluorinating a polymeric article to produce an article having surface fluorination; and (b) charging the fluorinated article in a manner sufficient to produce an electret. In one embodiment, the method includes charging the fluorinated article by contacting the fluorinated article with water in a manner sufficient to produce an electret, and drying the article. The method is useful for making the above-described electrets. In another embodiment, the method includes charging the fluorinated article by impinging jets of water or a stream of water droplets onto the fluorinated article at a pressure and for a period sufficient to produce an electret, and drying the article.

In other embodiments, the method includes fluorinating a polymeric article in the presence of an electrical discharge (e.g., an alternating current corona discharge at atmospheric pressure) to produce a fluorinated article. In one embodiment, the method includes fluorinating the polymeric article in an atmosphere that includes fluorine containing species selected from the group consisting of elemental fluorine, fluorocarbons, hydrofluorocarbons, fluorinated sulfur, fluorinated nitrogen and combinations thereof. Examples of suitable fluorine containing species include C_5F_{12}, C_2F_6, CF_4, hexafluoropropylene, SF_6, NF_3, and combinations thereof.

In other embodiments, the method includes fluorinating the polymeric article in an atmosphere that includes elemental fluorine.

In other embodiments, the method of making the electret includes: (A) fluorinating a nonwoven polymeric fibrous web (i) in an atmosphere that includes fluorine containing species and an inert gas, and (ii) in the presence of an electrical discharge to produce a web having surface fluorination; and (B) charging the fluorinated web in a manner sufficient to produce an electret.

In other aspects, the invention features a method of filtering that includes passing an aerosol through the above-described electrets to remove contaminants.

The fluorinated electrets of the invention exhibit a relatively high oily mist resistance relative to non-fluorinated electrets.

GLOSSARY

In reference to the invention, these terms having the meanings set forth below:

"electret" means a dielectric material exhibiting a quasi-permanent electrical charge. The term "quasi-permanent" means that the time constants characteristic for the decay of the charge are much longer than the time period over which the electret is used;

"surface modified" means that the chemical structure at the surface has been altered from its original state.

"surface fluorination" means the presence of fluorine atoms on a surface (e.g., the surface of an article);

"fluorine containing species" means molecules and moieties containing fluorine atoms including, e.g., fluorine atoms, elemental fluorine, and fluorine containing radicals;

"fluorinating" means placing fluorine atoms on the surface of an article by transferring fluorine containing species from a gaseous phase to the article by chemical reaction, sorption, condensation, or other suitable means;

"aerosol" means a gas that contains suspended particles in solid or liquid form; and

"contaminants" means particles and/or other substances that generally may not be considered to be particles (e.g., organic vapors).

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a plot of % DOP Penetration vs. DOP Load for Examples 36 and 37.

FIG. 2 is a plot of % DOP Penetration vs. DOP Load for Examples 38 and 39.

FIG. 3 is a plot of % DOP Penetration vs. DOP Load for Example 40.

DESCRIPTION OF PREFERRED EMBODIMENTS

The electret includes a surface modified polymeric article (e.g., a nonwoven polymeric fibrous web) produced by fluorinating a polymeric article. The electrets preferably have sufficient surface fluorination to provide oily mist resistance. One measure of oily mist resistance is how well the electret maintains its Quality Factor during challenge with an aerosol. The Quality Factor can be calculated from results obtained from the dioctylphthalate ("DOP") initial penetration test ("the DOP test"). The DOP test also provides a relative measure of the charge state of the filter. The DOP test procedure involves forcing DOP aerosol at a face velocity of 6.9 cm/second for a period of about 30 seconds through the sample, measuring the pressure drop across the sample (Pressure Drop measured in mmH_2O) with a differential manometer, and measuring the percent DOP penetration (DOPPen %). The Quality Factor (QF) (measured in $1/mmH_2O$) can be calculated from these values according to the following formula:

$$QF[1/\mathrm{mm}H_2O] = \frac{-\mathrm{Ln}\frac{DOP\,Penetration(\%)}{100}}{Pressure\,Drop[\mathrm{mm}\,H_2O]}$$

The higher the Quality Factor at a given flow rate, the better the filtering performance of the electret.

Preferred electrets have a Quality Factor of at least about $0.25/mmH_2O$, preferably at least about 0.5/mmH20, more preferably at least about $1.0/mmH_2O$.

Electron spectroscopy for chemical analysis ("ESCA") (also known as X-ray photoelectron spectroscopy ("XPS")) provides one measure of surface fluorination. Preferably the surface of the electret exhibits at least about 45 atomic % fluorine, more preferably at least about 50 atomic % fluorine when analyzed by ESCA. ESCA analyzes the elemental composition of the outermost surface (i.e., approximately 10 to 50 Å) of a specimen. ESCA can be used to detect all elements in the periodic table except helium and hydrogen.

The electret also has a CF_3:CF_2 ratio at the surface of the electret of at least about 0.25, preferably at least about 0.45, and more preferably greater than 0.9, as determined according to the Method For Determining CF_3:CF_2 ratio set forth in the Example section below.

In one embodiment, the electrets include nonwoven polymeric fibrous webs that include fibers such as, e.g., meltblown microfibers, staple fibers, fibrillated films, and combinations thereof. The fibers can be formed from resins. Preferably the resin is a thermoplastic nonconductive, i.e., having a resistivity of greater than 10^{14} ohm-cm, resin. The resin used to form the fibers should be substantially free of materials such as antistatic agents that could increase the electrical conductivity or otherwise interfere with the ability of the fibers to accept and hold electrostatic charges.

Examples of useful thermoplastic resins include polyolefins such as, e.g., polypropylene, polyethylene, poly-(4-methyl-1-pentene), and combinations thereof, halogenated vinyl polymers (e.g., polyvinyl chloride), polystyrene, polycarbonates, polyesters, and combinations thereof.

Additives can be blended with the resin including, e.g., pigment, UV stabilizers, antioxidants, and combinations thereof.

Meltblown microfibers can be prepared as described in Wente, Van A., "Superfine Thermoplastic Fibers," *Industrial Eng. Chemistry*, Vol. 48, pp. 1342–1346 and in Report No. 4364 of the Naval Research laboratories, published May 25, 1954, entitled, "Manufacture of Super Fine Organic Fibers," by Wente et al. Meltblown microfibers preferably have an effective fiber diameter in the range of less than 1 to 50 μm as calculated according to the method set forth in Davies, C. N., "The Separation of Airborne Dust and Particles," Institution of Mechanical Engineers, London, Proceedings 1B, 1952.

The presence of staple fibers provides a more lofty, less dense web than a web constructed solely of meltblown microfibers. Preferably the electret contains more than 70% by weight staple fibers. Webs containing staple fibers are disclosed in U.S. Pat. No. 4,118,531 (Hauser).

Electrets that include a nonwoven polymeric fibrous web preferably have a basis weight in the range of about 10 to 500 gm^2, more preferably about 10 to 100 gm^2. The thickness of the nonwoven polymeric fibrous web is preferably about 0.25 to 20 mm, more preferably about 0.5 to 2 mm.

The nonwoven polymeric webs of the electret can also include particulate matter as disclosed, for example, in U.S. Pat. No. 3,971,373, (Braun), U.S. Pat. No. 4,100,324 (Anderson), and U.S. Pat. No. 4,429,001 (Kolpin et al.).

Electret Preparation

The electrets can be prepared by fluorinating a polymeric article, optionally in the presence of a surface modifying electrical discharge, and charging the fluorinated article to produce an electret.

The fluorination process includes modifying the surface of the polymeric article to contain fluorine atoms by exposing the polymeric article to an atmosphere that includes fluorine containing species. The fluorination process can be performed at atmospheric pressure or under reduced pressure. The fluorination process is preferably performed in a controlled atmosphere to prevent contaminants from inter-

fering with the addition of fluorine atoms to the surface of the article. The atmosphere should be substantially free of oxygen and other contaminants. Preferably the atmosphere contains less than 0.1% oxygen.

The fluorine containing species present in the atmosphere can be derived from fluorinated compounds that are gases at room temperature, become gases when heated, or are capable of being vaporized. Examples of useful sources of fluorine containing species include, fluorine atoms, elemental fluorine, fluorocarbons (e.g., C_5F_{12}, C_2F_6, CF_4, and hexafluoropropylene), hydrofluorocarbons (e.g., CF_3H), fluorinated sulfur (e.g., SF_6), fluorinated nitrogen (e.g., NF_3), fluorochemicals such as e.g., CF_3OCF_3 and fluorochemicals available under the trade designation Fluorinert such as, e.g., Fluorinert FC-43 (commercially available from Minnesota Mining and Manufacturing Company, Minnesota), and combinations thereof.

The atmosphere of fluorine containing species can also include an inert diluent gas such as, e.g., helium, argon, nitrogen, and combinations thereof.

The electrical discharge applied during the fluorination process is capable of modifying the surface chemistry of the polymeric article when applied in the presence of a source of fluorine containing species. The electrical discharge is in the form of plasma, e.g., glow discharge plasma, corona plasma, silent discharge plasma (also referred to as dielectric barrier discharge plasma and alternating current ("AC") corona discharge), and hybrid plasma, e.g., glow discharge plasma at atmospheric pressure, and pseudo glow discharge. Preferably the plasma is an AC corona discharge plasma at atmospheric pressure. Examples of useful surface modifying electrical discharge processes are described in U.S. Pat. Nos. 5,244,780, 4,828,871, and 4,844,979.

Another fluorination process includes immersing a polymeric article into a liquid that is inert with respect to elemental fluorine, and bubbling elemental fluorine gas through the liquid to produce a surface fluorinated article. Examples of useful liquids that are inert with respect to fluorine include perhalogenated liquids, e.g., perfluorinated liquids such as Performance Fluid PF 5052 (commercially available from Minnesota Mining and Manufacturing Company). The elemental fluorine containing gas that is bubbled through the liquid can include an inert gas such as, e.g., nitrogen, argon, helium, and combinations thereof.

Charging the polymeric article to produce an electret can be accomplished using a variety of techniques, including, e.g., hydrocharging, i.e., contacting an article with water in a manner sufficient to impart a charge to the article, followed by drying the article, and DC corona charging. The charging process can be applied to one or more surfaces of the article.

One example of a useful hydrocharging process includes impinging jets of water or a stream of water droplets onto the article at a pressure and for a period sufficient to impart a filtration enhancing electret charge to the web, and then drying the article. The pressure necessary to optimize the filtration enhancing electret charge imparted to the article will vary depending on the type of sprayer used, the type of polymer from which the article is formed, the type and concentration of additives to the polymer, and the thickness and density of the article. Pressures in the range of about 10 to about 500 psi (69 to 3450 kPa) are suitable. An example of a suitable method of hydrocharging is described in U.S. Pat. No. 5,496,507 (Angadjivand et al.).

The jets of water or stream of water droplets can be provided by any suitable spray device. One example of a useful spray device is the apparatus used for hydraulically entangling fibers.

Examples of suitable DC corona discharge processes are described in U.S. Pat. No. 30,782 (van Turnhout), U.S. Pat. No. 31,285 (van Turnhout), U.S. Pat. No. 32,171 (van Turnhout), U.S. Pat. No. 4,375,718 (Wadsworth et al.), U.S. Pat. No. 5,401,446 (Wadsworth et al.), U.S. Pat. No. 4,588,537 (Klasse et al.), and U.S. Pat. No. 4,592,815 (Nakao).

The fluorinated electrets formed by the methods described herein are suitable for use as, e.g., electrostatic elements in electro-acoustic devices such as microphones, headphones and speakers, fluid filters, dust particle control devices in, e.g., high voltage electrostatic generators, electrostatic recorders, respirators (e.g., prefilters, canisters and replaceable cartridges), heating, ventilation, air conditioning, and face masks.

The invention will now be described further by way of the following examples.

EXAMPLES

Test Procedures

Test procedures used in the examples include the following.

Method for Determining CF_3:CF_2

ESCA data was collected on a PHI 5100 ESCA system (Physical Electronics, Eden Prairie, Minn.) using a non-monochromatic MgKα x-ray source and a 45 degree electron takeoff angle with respect to the surface. The carbon (1s) spectra were peak fit using a nonlinear least-squares routine supplied by PHI (Physical Electronics, Eden Prairie, Minn.). This routine used a linear background subtraction, and a gaussian peak save for the component peaks. The spectra were referenced to the hydrocarbon peak at 285.0 eV. The CF_3 and CF_2 components were identified as the peaks located at about 294 eV and 292 eV respectively (according to the procedure described in Strobel et al., J. Polymer Sci. A: Polymer Chemistry, Vol. 25, pp. 1295–1307 (1987)). The CF_3:CF_2 ratio represent the ratio of the peak areas of the CF_3 and CF_2 components.

Initial Dioctylphthalate Penetration (DOP) and Pressure Drop Test Procedure

Initial DOP penetration is determined by forcing 0.3 micrometer diameter dioctyl phthalate (DOP) particles at a concentration of between 70 and 140 mg/m^3 (generated using a TSI No. 212 sprayer with four orifices and 30 psi clean air) through a sample of filter media which is 4.5 inches in diameter at a rate of 42.5 L/min (a face velocity of 6.9 centimeters per second). The sample is exposed to the DOP aerosol for 30 seconds until the readings stabilize. The penetration is measured with an optical scattering chamber, Percent Penetration Meter Model TPA-8F available from Air Techniques Inc.

Pressure drop across the sample is measured at a flow rate of 42.5 L/min (a face velocity of 6.9 cm/sec) using an electronic manometer. Pressure drop is reported in mm of water ("mm H_2O").

DOP penetration and pressure drop are used to calculate the quality factor "QF" from the natural log (ln) of the DOP penetration by the following formula:

$$QF[1/\mathrm{mm\,H_2O}] = \frac{-\ln\left(\frac{DOP\,Penetration(\%)}{100}\right)}{Pressure\,Drop[\mathrm{mm\,H_2O}]}$$

A higher initial QF indicates better initial filtration performance. A decreased QF effectively correlates with decreased filtration performance.

DOP Loading Test

DOP loading is determined using the same test equipment used in the DOP penetration and pressure drop tests. The test

sample is weighed and then exposed to the DOP aerosol for at least 45 min to provide a minimum exposure of at least about 130 mg. DOP penetration and pressure drop are measured throughout the test at least as frequently as once per minute. The mass of DOP collected is calculated for each measurement interval from the measured penetration, mass of the filter web, and total mass of DOP collected on the filter web during exposure ("DOP Load").

Corona Fluorination

Example 1

A blown polypropylene microfiber web prepared from Exxon 3505G polypropylene resin (Exxon Corp.) and having an effective fiber diameter of 7.5 μm and a basis weight of 62 g/m^2 was prepared as described in Wente, Van A., "Superfine Thermoplastic Fibers," *Industrial Eng. Chemistry*, Vol. 48, pp. 1342–1346.

The blown microfiber web was then AC corona fluorinated in a 1% by volume C_2F_6 in helium atmosphere at a corona energy of 34 J/cm^2, which corresponded to a corona power of 2000 W at a substrate speed of 1 m/min. The AC corona fluorination treatment was performed in an AC corona system that included the so-called "double-dielectric" electrode configuration with a ground roll consisting of 40 cm diameter nickel-plated aluminum roll covered with 1.5 mm of poly(ethylene terephthalate) and maintained at a temperature of 23° C. using recirculating, pressurized water. The powered electrodes consisted of 15 individual ceramic-covered electrodes (available from Sherman treaters Ltd., Thame, United Kingdom) each with a 15 mm square cross-section and an active length of 35 cm. The electrodes were connected to a model RS48-B (4 kW) variable-frequency power supply (available from ENI Power Systems Inc., Rochester, N.Y.). The net power dissipated in the AC corona was measured with a directional power meter incorporated into the ENI supply. The frequency of the output power was manually adjusted to about 16 kHz to obtain optimal impedance matching (minimum reflected power).

The AC corona system was enclosed within a controlled environment. Prior to treatment, the atmosphere surrounding the AC corona treatment system was purged with helium, and then continually flushed with 100 liters/min of 1% by volume C_2F_6 in helium, which was introduced near the electrodes.

The microfiber web was taped onto a carrier film of 0.05 mm thick bi-axially-oriented polypropylene (BOPP), and then placed on the ground roll such that the carrier film was in contact with the ground roll, causing one side of the blown microfiber web to be exposed to the discharge. After treatment, the blown microfiber web was flipped over, retaped to the carrier film, and AC corona treated a second time under the same conditions as the first treatment to expose the other side of the blown microfiber web to the discharge.

Example 2

A G100 Filtrete fibrillated film web (available from Minnesota Mining and Manufacturing), having a basis weight of 100 g/m^2, was corona fluorinated following the method described in Example 1, with the exception that the ground roll was maintained at a temperature of 25° C.

Example 3

A polyethylene meltblown microfiber web, prepared from Aspun PE-6806 polyethylene resin (DOW Chemical Company, Michigan) and having a basis weight of 107 g/m^2, was corona fluorinated following the method described in Example 2.

Example 4

A polyester staple fiber web (available from Rogers Corporation), having a basis weight of 200 g/m^2, was corona fluorinated following the method described in Example 2.

Example 5

A poly-4-methyl-1-pentene meltblown microfiber web prepared from TPX MX-007 poly-4-methyl-1-pentene resin (Mitsui), and having a basis weight of 50 g/m^2 and an effective fiber diameter of 8.1 μm, was corona fluorinated following the method described in Example 2.

Examples 6–9

Examples 6–9 were prepared following the procedure in Example 1 except that the source of fluorine containing species was as follows: 1% CF_4 (Example 6), and 0.1% hexafluoropropylene (Example 7), 0.1% C_5F_{12} (Example 8), and 1.0% C_5F_{12} (Example 9).

The surface chemistry of each of the sample webs of Examples 1–9 was determined by ESCA analysis using a PHI 5100 ESCA system. The CF_3:CF_2 ratio was determined for each of the samples of Examples 1–9 from the ESCA data according to the above-described method. The results are reported in atomic % in Table I.

TABLE I

Example	Carbon	Nitrogen	Oxygen	Fluorine	CF_3:CF_2
1	43		5.7	51	1.09
2	44		6.2	50	1.37
3	49	0.2	8.2	42	1.10
4	42	0.5	7.8	49	0.99
5	44	0.0	2.9	53	1.19
6	41		3.5	55	0.86
7	41		2.7	56	0.97
8	42		6.4	52	0.91
9	43		5.2	51	0.89

Hydrocharging

Example 10

A fluorinated polypropylene blown microfiber web prepared as described above in Example 1, was passed over a vacuum slot at a rate of 5 cm/sec centimeters/second) while deionized water was sprayed onto the web at a hydrostatic pressure of about 90 psi from a pair of Spraying Systems Teejet 9501 sprayer nozzles mounted 10 cm apart and centered 7 cm above the vacuum slot. The sample was then inverted and passed through the deionized water spray a second time such that both sides of the web were sprayed with water. The deionized water spray was then removed and the web was again passed over the vacuum slot to remove excess water. The web was then hung to dry at ambient conditions.

Example 11

A fluorinated poly-4-methyl1-pentene meltblown microfiber web prepared according to Example 5 was charged following the procedure of Example 10.

Examples 10A–11A

Examples 10A–11A were prepared following the procedures of Example 10 and 11 respectively, with the exception

that, after corona fluorination and prior to hydrocharging, each of the fluorinated webs of Examples 10A–11A were subjected to an anneal at 140° C. (300° F.) for about 10 minutes.

Examples 13, 15, 16, 18 and 20

Examples 13, 15, 16, 18 and 20 were charged following the procedure of Example 10, with the exception that the fluorinated polymeric fibrous webs used in each of Examples 13, 15, 16, 18 and 20 were as follows: a fluorinated polyethylene microfiber web prepared according to Example 3 above (Example 13); a fluorinated polyester staple fiber web prepared according to Example 4 (Example 15); a fluorinated G100 Filtrete fibrillated film web prepared according to Example 2 (Example 16); a fluorinated polypropylene needle punched web (12 denier/fiber fibers of Exxon 3505 polypropylene resin), having a basis weight of about:200 gm^2, and having been corona fluorinated following the method described in Example 1 (Example 18); and a polypropylene melt blown fine fiber web, having a basis weight of 46 gm^2 and an effective fiber diameter of 3.7 μm, and having been corona fluorinated following the method described in Example 1 with the exception that 0.2% C_5F_{12} was used instead of 1% C_2F_6 (Example 20).

DC Corona Charging

Example 12

The fluorinated polyethylene meltblown microfiber web of Example 3 was charged using a DC corona discharge as follows. The fluorinated web was placed in contact with an aluminum ground plane, and then passed under an electrically positive DC corona source, in air, at a rate of about 1.2 meters/min, while maintaining a current to ground plane of about 0.01 mA/cm of corona source length. The distance from corona source to ground was about 4 cm.

Examples 14, 17, 19

Examples 14, 17 and 19 were charged following the procedure of Example 12, with the exception that the fluorinated polymeric fibrous webs for each of Examples 14, 17 and 19 were as follows: a fluorinated polyester staple fiber web prepared following the procedure of Example 4 (Example 14); a fluorinated polypropylene needle punched web (12 denier/fiber fibers made from Exxon 3505 polypropylene resin), having a basis weight of about 200 gm^2, and having been corona fluorinated following the method described in Example 1 (Example 17); and a fluorinated polypropylene meltblown fine fiber web, having a basis weight of 46 g/m^2 and an effective fiber diameter of 3.7 μm, and having been corona fluorinated following the method described in Example 1 with the exception that 0.2% C_5F_{12} was used instead of 1% C_2F_6 (Example 19).

Example 21–35

Examples 21–35 were prepared by fluorinating polypropylene blown microfiber webs following the procedure of Example 1, with the exception that the source of fluorine for each of Examples 21–35 was as follows: 1% CF_4 (Examples 21–23), 1% C_2F_6 (Examples 24–26), 0.1% hexafluoropropylene (Examples 27–29), 0.1% C_5F_{12} (Examples 30–32), and 1.0% C_5F_{12} (Examples 33–35).

The fluorinated webs of Examples 23, 26, 29, 32, and 35 were then charged following the hydrocharging process described above in Example 10.

The fluorinated webs of Examples 22, 25, 28, 31 and 34 were then charged following the DC corona charging process described above in Example 12.

% DOP penetration ("%DOP PEN"), Pressure Drop (mmH_2O), and the Quality Factor ("QF") for each of the electrets of Examples 10–35 were determined according to the above-described Initial DOP Penetration and Pressure Drop Test Procedure. The results are summarized in Table II.

TABLE II

EXAMPLE	% DOP PEN	PRESSURE DROP	QF
10	0.119	3.65	1.84
10A	0.140	3.21	2.05
11	2.45	1.46	2.54
11A	0.778	1.60	3.04
12	56.1	1.14	0.51
13	38.1	1.15	0.84
14	78.3	0.38	0.64
15	65.6	0.41	1.03
16	27.3	0.40	3.25
17	70.4	0.19	1.85
18	37.6	0.19	5.15
19	0.81	10.58	0.46
20	0.006	11.3	0.86
21	55.6	2.83	0.21
22	15.0	3.28	0.58
23	0.288	3.09	1.89
24	54.1	3.05	0.20
25	14.3	3.32	0.59
26	0.243	3.08	1.95
27	59.0	2.81	0.19
28	16.2	2.80	0.65
29	0.276	2.90	2.03
30	52.5	3.15	0.20
31	14.0	3.11	0.63
32	0.250	2.99	2.00
33	45.3	3.10	0.26
34	14.9	2.93	0.65
35	0.244	3.14	1.92

Examples 36–39

Four fluorinated, polypropylene microfiber webs were prepared according to Example 1 with the exception that the source of fluorine containing species was as follows: 0.1% hexafluoropropylene ("HFP") (Examples 36 and 38) and 0.1% C_5F_{12} (Example 37 and 39).

Examples 36 and 37 further included charging the fluorinated polypropylene webs following the hydrocharging charging procedure of Example 10.

Examples 38 and 39 further included charging the fluorinated polypropylene webs following the DC corona charging procedure of Example 12.

Examples 36–39 were subjected to the above-described DOP Loading Test. The % DOP Penetration versus DOP loading (the amount of DOP collected on the web in grams) for each of Examples 36–39 was measured according to the above-described DOP Loading Test Procedure. The resulting data are plotted as % DOP penetration versus DOP load (grams) in FIGS. 1 and 2 as follows: Examples 36 and 37 (indicated with x's and solid circles respectively) (FIG. 1), and Examples 38 and 39 (indicated with x's and solid circles respectively) (FIG. 2).

Example 40

A 7 in. sample of polypropylene microfiber web having a basis weight 61 g/m^2 was placed under a nitrogen atmosphere. A gaseous mixture of 5% by volume elemental fluorine diluted in nitrogen was passed through the polypropylene microfiber web at a rate of 1.0 l/min for 10 minutes. The fluorine concentration was then increased to 10% by volume diluted in nitrogen and passed through the web at a rate of 1.0 l/min for an additional 20 minutes.

The sample was then analyzed by ESCA and determined to have 62 atomic % fluorine and a CF_3:CF_2 ratio of 0.59, as determined according to the above-described Method for Determining CF_3:CF_2.

The sample was then charged using a DC corona discharge as described above in Example 12, and subjected to the above-described DOP Loading Test. The resulting data are plotted as % DOP Penetration versus DOP Load (grams) in FIG. 3.

Example 41

A polypropylene blown microfiber web, having a basis weight of 20 g/m^2 and a web width of 15 cm, was vacuum glow-discharge treated in a C_5F_{12} environment. The glow-discharge treatment was performed in a vacuum chamber. The vacuum chamber contained a roll-to-roll glow discharge system consisting of an unwind roller, glow discharge electrodes, and a windup roller for the continuous treatment of the blown microfiber web. Two stainless steel electrodes were in the parallel plate configuration, each electrode was 20 cm wide and 33 cm long and they were separated by a gap of 2.5 cm. The top electrode was grounded and the bottom electrode was powered by a 13.56 MHz rf generator (Plasma-Therm). The web traveled between the two electrodes and in contact with the top, grounded electrode so that one side of the web was exposed to the discharge.

After loading the roll of blown microfiber web onto the unwind roller under C_5F_{12} vapor at a pressure of 0.1 Torr. The blown microfiber web was advanced through the electrodes at a speed of 17 cm/min to achieve an exposure time to the plasma of 2 minutes. The discharge power was 50 W. After the first side was treated, the chamber was vented and the web roll replaced onto the unwind roller to allow the other side of the web to be treated. The treatment of the second side of the web occurred under the same conditions as the first side. After the fluorination, Example 41 was DC-corona charged following the process described above in Example 12.

% DOP Penetration ("%DOP PEN") for Example 41 was determined according to the above-described Initial DOP Penetration and Pressure Drop Test Procedure. The results are summarized in Table III.

TABLE III

Loading Time (min)	% DOP Penetration Example 14
0.5	28
10	28

Other embodiments are within the following claims. Although the electret has been described reference to nonwoven polymeric fibrous webs, the electret can be a variety of polymeric articles including, e.g., those polymeric articles described in U.S. patent application Ser. No. 09/106,506 entitled, "Structured Surface Filter Media," (Insley et al.), filed on Jun. 18, 1998.

All of the patents and patent applications cited above are incorporated by reference into this document in total.

What is claimed is:

1. A method of making an electret comprising:

fluorinating a polymeric nonwoven web to produce an article having surface fluorination; and

charging the fluorinated web in a manner sufficient to produce an electret,

the electret comprising at least about 45 atomic % fluorine as detected by ESCA.

2. The method of claim 1, comprising charging the fluorinated article by contacting the fluorinated article with water in a manner sufficient to produce an electret, and drying the article.

3. The method of claim 1, comprising charging the fluorinated article by impinging jets of water or a stream of water droplets onto the fluorinated article at a pressure and for a period sufficient to produce an electret, and drying the article.

4. The method of claim 1, comprising fluorinating the polymeric nonwoven web in the presence of an electrical discharge to produce a fluorinated article.

5. The method of claim 4, comprising fluorinating the polymeric nonwoven web in the presence of an alternating current corona discharge at atmospheric pressure.

6. The method of claim 4, comprising fluorinating the polymeric nonwoven web in an atmosphere comprising fluorine containing species selected from the group consisting of elemental fluorine, fluorocarbons, hydrofluorocarbons, fluorinated sulfur, fluorinated nitrogen or a combination thereof.

7. The method of claim 6, wherein the fluorine containing species are selected from the group consisting of C_5F_{12}, C_2F_6, CF_4, hexafluoropropylene, SF_6, NF_3, or a combination thereof.

8. The method of claim 1, comprising fluorinating the polymeric nonwoven web in an atmosphere comprising elemental fluorine.

9. The method of claim 1, wherein the electret comprises a CF_3:CF_2 ratio of at least about 0.25 as determined according to the Method for Determining CF_3:CF_2.

10. The method of claim 1, wherein the electret comprises a CF_3:CF_2 ratio of at least about 0.45 as determined according to the Method for Determining CF_3:CF_2.

11. The method of claim 1, wherein the electret has a Quality Factor of at least about 0.25/mmH_2O.

12. The method of claim 1, comprising charging the fluorinated article with a direct current corona discharge to produce an electret.

13. The method of claim 1, further comprising annealing the fluorinated article prior to charging the fluorinated article.

14. The method of claim 1, wherein the electret comprises at least about 50 atomic % fluorine as detected by ESCA.

15. The method of claim 1, wherein the charged nonwoven web has surface fluorination that comprises CF_3 and CF_2 at a CF_3:CF_2 ratio of at least 0.45 as determined according to the method for determining CF_3:CF_2.

16. The method of claim 1, wherein the charged nonwoven web has surface fluorination that comprises CF_3 and CF_2 at a CF_3:CF_2 ratio of greater than 0.9 as determined according to the method for determining CF_3:CF_2.

17. A method of making an electret comprising:

a) fluorinating a nonwoven polymeric fibrous web

i) in an atmosphere comprising fluorine containing species and an inert gas, and

ii) in the presence of an electrical discharge to produce a web having surface fluorination; and

b) charging the fluorinated web in a manner sufficient to produce an electret,

the electret comprising at least about 45 atomic % fluorine as detected by ESCA.

18. A method of making an electret article, which method comprises the steps of:

(a) providing a nonwoven fibrous web, wherein the fibers comprise a polymeric nonconductive thermoplastic resin;

(b) placing fluorine atoms on the surface of the polymeric fibers by transferring a fluorine-containing species from a gaseous phase to the fibers; and

(c) charging the fluorinated nonwoven fibrous web,

the charged nonwoven web comprising at least about 45 atomic % fluorine as detected by ESCA.

19. The method of claim 18, wherein the fibers are fluorinated in the presence of a surface modifying electrical discharge.

20. The method of claim 19, wherein the gaseous phase includes an inert gas, the fluorine-containing species being present in the inert gas.

21. The method of claim 20, wherein the fluorination step is performed at atmospheric pressure.

22. The method of claim 20, wherein the fluorination step is performed at reduced pressure.

23. The method of claim 18, wherein the fluorination step is performed in a controlled atmosphere that prevents contaminants from interfering with the addition of fluorine atoms to the surface of the polymeric fibers.

24. The method of claim 23, wherein the controlled atmosphere is substantially free of oxygen and other contaminants.

25. The method of claim 24, wherein the controlled atmosphere contains less than 0.1 percent oxygen.

26. The method of claim 18, wherein the fluorine-containing species includes one or more of the following: fluorine atoms, elemental fluorine, fluorocarbons, hydrofluorocarbons, fluorinated sulfur, fluorinated nitrogen, CF_3OCF_3, and combinations thereof.

27. The method of claim 26, wherein the inert gas is helium, argon, nitrogen, or a combination thereof.

28. The method of claim 19, wherein the surface modifying electrical discharge comprises the use of plasma.

29. The method of claim 18, wherein the fluorination step includes bubbling elemental fluorine gas through a liquid.

30. The method of claim 18, wherein the charging step includes hydrocharging, DC corona discharge, or a combination thereof.

31. The method of claim 30, wherein the charging step includes hydrocharging.

32. The method of claim 31, wherein the charging step includes DC corona discharge.

33. The method of claim 18, wherein the fibers are microfibers.

34. The method of claim 33, wherein the fibers are melt-blown microfibers that have an effective fiber diameter of 1 to 50 micrometers.

35. The method of claim 34, wherein the melt melt-blown microfibers comprise polypropylene resin and have a resistivity of greater than 10^{14} ohm*cm.

36. The method of claim 35, wherein the nonwoven web has a basis weight of 10 to 100 g/m^2 and has a thickness of 0.25 to 20 mm.

37. The method of claim 18, wherein the charged nonwoven web has a surface fluorination that comprises CF_3 and CF_2 at a CF_3:CF_2 ratio of at least 0.45 as determined according to the method for determining CF_3:CF_2.

38. The method of claim 37, wherein the nonwoven web comprises polymeric microfibers.

39. The method of claim 38, wherein the charged nonwoven web exhibits a Quality Factor of at least about 1.0 per mm H_2O.

40. The method of claim 37, wherein the charged nonwoven web has a CF_3:CF_2 ratio of at least 0.9.

41. The method of claim 38, wherein the microfibers are melt-blown microfibers that have an effective fiber diameter of 1 to 50 μm.

42. The method of claim 41, wherein the microfibers are made from a resin that has a resistivity greater than 10^{14} ohm-cm.

43. The method of claim 42, wherein the microfibers comprise a thermoplastic polyolefin.

44. The method of claim 43, wherein the microfibers comprise polypropylene.

45. The method of claim 44, wherein the nonwoven web has a basis weight of 10 to 100 g/m^2.

46. The method of claim 45, wherein the nonwoven web has a thickness of 0.25 to 20 mm.

47. The method of claim 46, wherein the nonwoven web has been fluorinated by transferring a fluorine containing species from a gaseous phase to the nonwoven web.

48. A method of making an electret comprising:

fluorinating a polymeric article to produce an article having surface fluorination;

annealing the fluorinated article; and

charging the fluorinated article in a manner sufficient to produce an electret.

49. A method of making an electret comprising:

fluorinating a polymeric article in the presence of an alternating current corona discharge at atmospheric pressure to produce an article having surface fluorination; and

charging the fluorinated article in a manner sufficient to produce an electret.

50. A method of making an electret comprising:

fluorinating a polymeric article at atmospheric pressure to produce an article having surface fluorination; and

charging the fluorinated article in a manner sufficient to produce an electret.

51. A method of making an electret comprising:

fluorinating a polymeric article to produce an article having surface fluorination; and

charging the fluorinated article in a manner sufficient to produce an electret,

the electret having a quality factor of at least 1.0/mmH_2O.

52. The method of claim 51, wherein the electret has a Quality Factor of at least 1.5/mmH_2O.

53. The method of claim 51, wherein the electret has a Quality Factor of at least 2/mmH_2O.

54. The method of claim 51, wherein the electret has a Quality Factor of at least 3/mmH_2O.

55. The method of claim 51, wherein the electret has a Quality Factor of at least 5/mmH_2O.

56. A method of making an electret article, which method comprises the steps of:

(a) providing a nonwoven fibrous web, wherein the fibers comprise a polymeric nonconductive thermoplastic resin;

(b) placing fluorine atoms on the surface of the polymeric fibers by transferring a fluorine-containing species from a gaseous phase to the fibers; and

(c) charging the fluorinated nonwoven fibrous web,

the charged nonwoven web having a surface fluorination that comprises CF_3 and CF_2 at a CF_3:CF_2 ratio of at least 0.45 as determined according to the method for determining CF_3:CF_2.

57. A method of making an electret article, which method comprises the steps of:

(a) providing a nonwoven fibrous web, wherein the fibers comprise a polymeric nonconductive thermoplastic resin;

(b) placing fluorine atoms on the surface of the polymeric fibers by transferring a fluorine-containing species from a gaseous phase to the fibers; and

(c) charging the fluorinated nonwoven fibrous web,

the charged nonwoven web exhibiting a Quality Factor of at least about 1.0/mmH_2O.

58. A method of making an electret article, which method comprises the steps of:

(a) providing a nonwoven fibrous web, wherein the fibers comprise a polymeric nonconductive thermoplastic resin;

(b) bubbling elemental fluorine gas through a liquid;

(c) placing fluorine atoms on the surface of the polymeric fibers by transferring a fluorine-containing species from a gaseous phase to the fibers; and

(d) charging the fluorinated nonwoven fibrous web.

* * * * *

UNITED STATES PATENT AND TRADEMARK OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 6,397,458 B1
DATED : June 4, 2002
INVENTOR(S) : Jones, Marvin E.

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

Column 3,
Line 64, "0.5/mmH2O" should read as -- 0.5/mmH_2O --.

Column 4,
Line 49, both occurrences of "gm^2" should read as -- g/m^2 --.

Column 9,
Line 18, "about:200" should read as -- about 200 --.
Lines 21 and 45, "gm^2" should read as -- g/m^2 --.

Column 10,
Line 60, after "in." insert -- by 7in. --.

Signed and Sealed this

Third Day of December, 2002

JAMES E. ROGAN
Director of the United States Patent and Trademark Office

UNITED STATES PATENT AND TRADEMARK OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 6,397,458 B1
DATED : June 4, 2002
INVENTOR(S) : Jones et al.

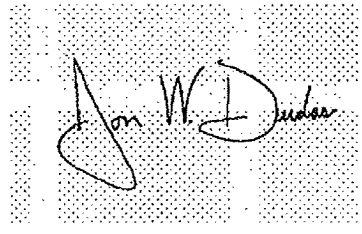
It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

Column 6,
Line 29, "save" should read -- shape --.

Column 13,
Line 23, between "of" and "fluorine" insert -- the --.
Line 46, "31" should read -- 18 --.
Line 55, "ohm*cm." should read -- ohm·cm. --.

Signed and Sealed this

Twentieth Day of December, 2005



JON W. DUDAS
Director of the United States Patent and Trademark Office

TAB 7

US006808551B2

(12) **United States Patent**
Jones et al.

(10) **Patent No.: US 6,808,551 B2**
(45) **Date of Patent: Oct. 26, 2004**

(54) **METHOD OF USING FLUORINATED ELECTRETS**

(75) Inventors: **Marvin E. Jones**, Grant Township, MN (US); **Christopher S. Lyons**, St. Paul, MN (US); **David B. Redmond**, St. Paul, MN (US); **Jeffrey L. Solomon**, Vadnais Heights, MN (US); **Seyed Abolhassan Angadjivand**, Woodbury, MN (US)

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(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: **10/681,670**

(22) Filed: **Oct. 7, 2003**

(65) **Prior Publication Data**

US 2004/0065196 A1 Apr. 8, 2004

Related U.S. Application Data

(63) Continuation of application No. 10/391,240, filed on Mar. 18, 2003, now Pat. No. 6,660,210, which is a continuation of application No. 10/126,028, filed on Apr. 19, 2002, now Pat. No. 6,562,112, which is a continuation of application No. 09/109,497, filed on Jul. 2, 1998, now Pat. No. 6,432,175.

(51) **Int. Cl.**[7] **B03C 3/28**
(52) **U.S. Cl.** **95/59**; 55/528; 55/DIG. 39; 96/69
(58) **Field of Search** ..... 95/57, 59; 96/15, 96/66–69; 55/528, DIG. 39

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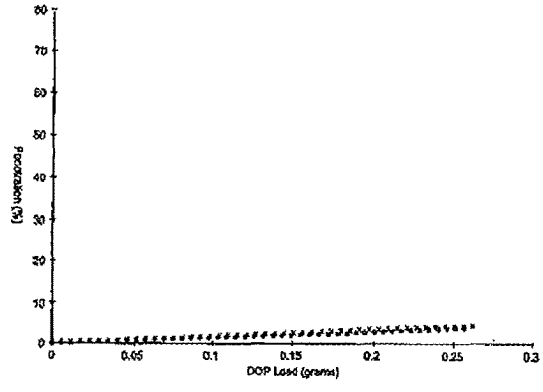
Primary Examiner—Richard L. Chiesa
(74) *Attorney, Agent, or Firm*—Karl Hanson; Allison Johnson

(57) **ABSTRACT**

An electret is described that includes a surface modified polymeric article having surface fluorination produced by fluorinating the polymeric article.

37 Claims, 3 Drawing Sheets



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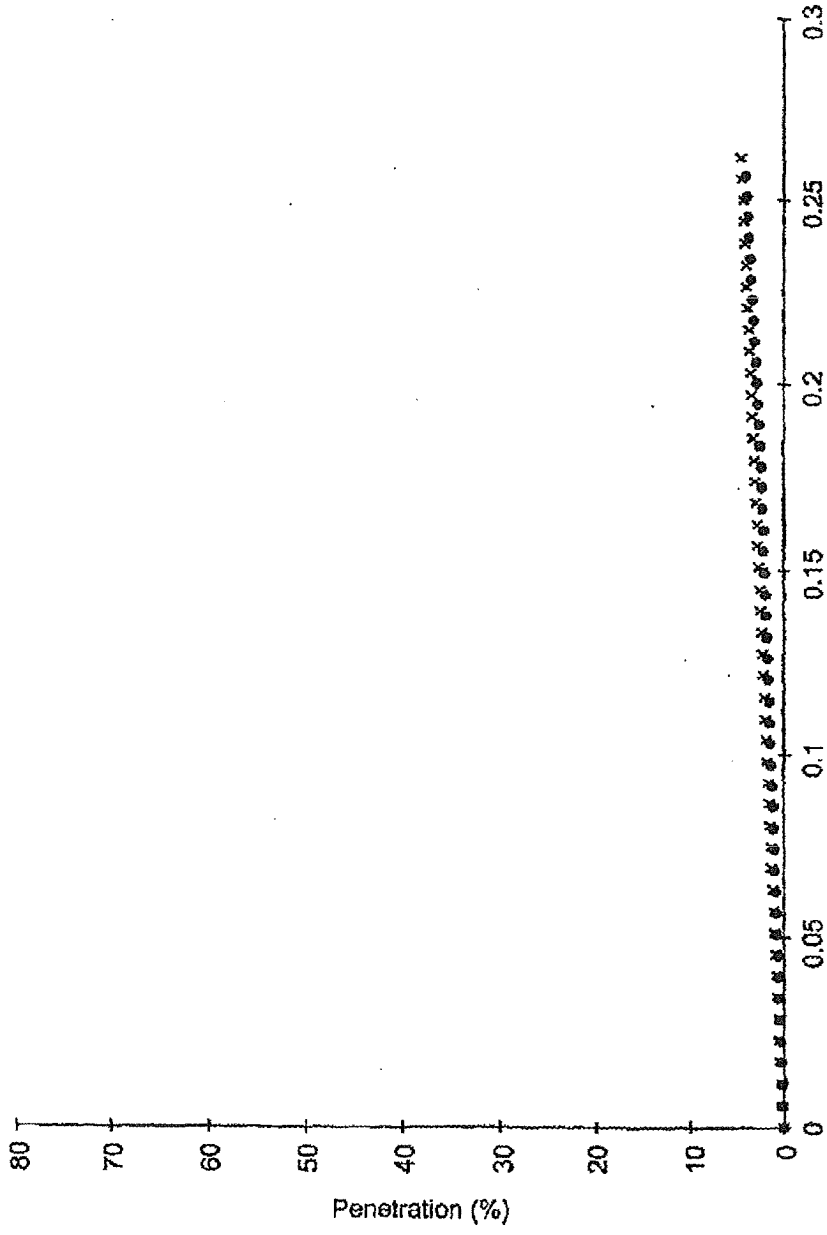
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0.25
0.2
0.15
0.1
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DOP Load (grams)
80
70
60
50
40
30
20
10
0
Penetration (%)

Fig. 1

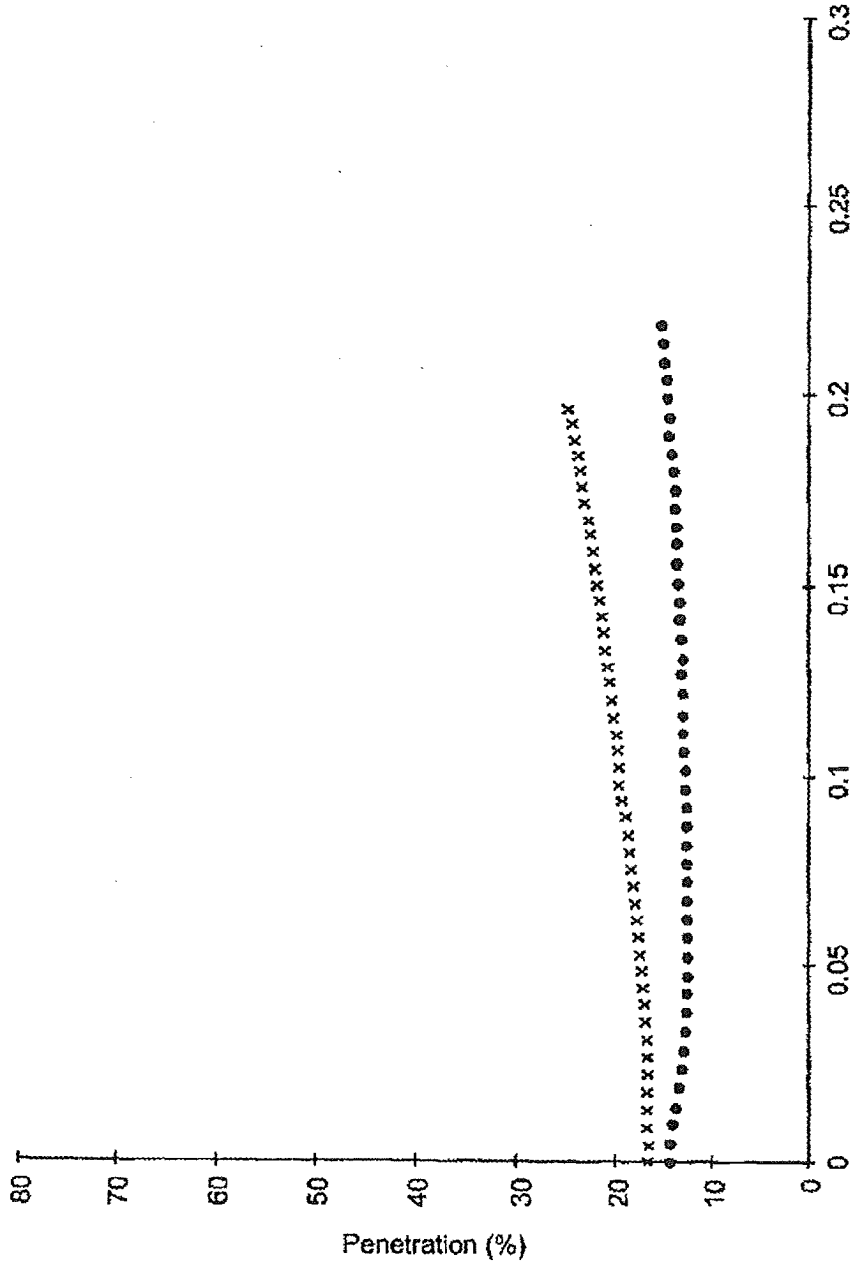

Penetration (%)
80
70
60
50
40
30
20
10
0
DOP Load (grams)
0
0.05
0.1
0.15
0.2
0.25
0.3

Fig. 2

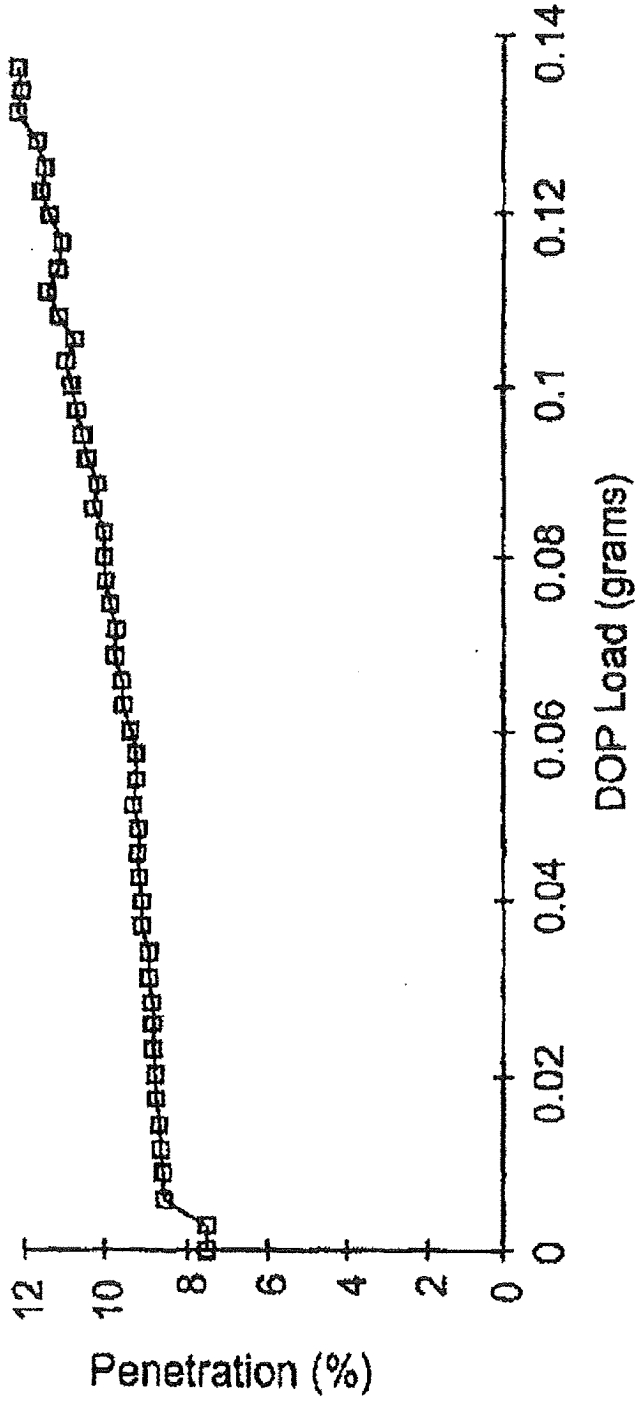

0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
DOP Load (grams)
12
10
8
6
4
2
0
Penetration (%)

Fig. 3

METHOD OF USING FLUORINATED ELECTRETS

CROSS-REFERENCE TO RELATED APPLICATIONS

This is a continuation of U.S. application Ser. No. 10/391,240, filed Mar. 18, 2003, now U.S. Pat. No. 6,660,210, which is a continuation of U.S. application Ser. No. 10/126,028, filed Apr. 19, 2002, now U.S. Pat. No. 6,562,112, which is a continuation of U.S. application Ser. No. 09/109,497, filed Jul. 2, 1998 now U.S. Pat. No. 6,432,175.

This invention relates to preparing fluorinated electrets.

BACKGROUND

The filtration properties of nonwoven polymeric fibrous webs can be improved by transforming the web into an electret, i.e., a dielectric material exhibiting a quasi-permanent electrical charge. Electrets are effective in enhancing particle capture in aerosol filters. Electrets are useful in a variety of devices including, e.g., air filters, face masks, and respirators, and as electrostatic elements in electro-acoustic devices such as microphones, headphones, and electrostatic recorders.

Electrets are currently produced by a variety of methods including direct current ("DC") corona charging (see, e.g., U.S. Pat. No. Re. 30,782 (van Turnhout)), and hydrocharging (see, e.g., U.S. Pat. No. 5,496,507 (Angadjivand et al.)), and can be improved by incorporating fluorochemicals into the melt used to produce the fibers of some electrets (see, e.g., U.S. Pat. No. 5,025,052 (Crater et al.)).

Many of the particles and contaminants with which electret filters come into contact interfere with the filtering capabilities of the webs. Liquid aerosols, for example, particularly oily aerosols, tend to cause electret filters to lose their electret enhanced filtering efficiency (see, e.g., U.S. Pat. No. 5,411,576 (Jones et al.)).

Numerous methods have been developed to compensate for loss of filtering efficiency. One method includes increasing the amount of the nonwoven polymeric web in the electret filter by adding layers of web or increasing the thickness of the electret filter. The additional web, however, increases the breathing resistance of the electret filter, adds weight and bulk to the electret filter, and increases the cost of the electret filter. Another method for improving an electret filter's resistance to oily aerosols includes forming the electret filter from resins that include melt processable fluorochemical additives such as fluorochemical oxazolidinones, fluorochemical piperazines, and perfluorinated alkanes. (See, e.g., U.S. Pat. No. 5,025,052 (Crater et al.)). The fluorochemicals should be melt processable, i.e., suffer substantially no degradation under the melt processing conditions used to form the microfibers that are used in the fibrous webs of some electrets. (See, e.g., WO 97/07272 (Minnesota Mining and Manufacturing)).

SUMMARY OF THE INVENTION

In one aspect, the invention features an electret that includes a surface modified polymeric article having surface fluorination produced by fluorinating a polymeric article.

In one embodiment, the article includes at least about 45 atomic % fluorine as detected by ESCA. In another embodiment, the article includes a CF_3:CF_2 ratio of at least about 0.25 as determined according to the Method for Determining CF_3:CF_2. In other embodiments, the article includes a CF_3:CF_2 ratio of at least about 0.45 as determined according to the Method for Determining CF_3:CF_2.

In one embodiment, the article has a Quality Factor of at least about 0.25/mmH_2O, (preferably at least about 0.5/mmH_2O, more preferably at least about 1/mmH_2O).

In some embodiments, the article includes a nonwoven polymeric fibrous web. Examples of suitable fibers for the nonwoven polymeric fibrous web include polycarbonate, polyolefin, polyester, halogenated polyvinyl, polystyrene, and combinations thereof. Particularly useful fibers include polypropylene, poly-(4-methyl-1-pentene), and combinations thereof. In one embodiment, the article includes melt-blown microfibers.

In another aspect, the invention features an electret that includes a polymeric article having at least about 45 atomic % fluorine as detected by ESCA, and a CF_3:CF_2 ratio of at least about 0.45 as determined according to the Method for Determining CF_3:CF_2. In another embodiment, the electret includes at least about 50 atomic % fluorine as detected by ESCA, and a CF_3:CF_2 ratio of at least about 0.25 as determined according to the Method for Determining CF_3:CF_2.

In other aspects, the invention features a respirator that includes the above-described electrets. In still other aspects, the invention features a filter that includes the above-described electrets.

In one aspect, the invention features a method of making an electret that includes: (a) fluorinating a polymeric article to produce an article having surface fluorination; and (b) charging the fluorinated article in a manner sufficient to produce an electret. In one embodiment, the method includes charging the fluorinated article by contacting the fluorinated article with water in a manner sufficient to produce an electret, and drying the article. The method is useful for making the above-described electrets. In another embodiment, the method includes charging the fluorinated article by impinging jets of water or a stream of water droplets onto the fluorinated article at a pressure and for a period sufficient to produce an electret, and drying the article.

In other embodiments, the method includes fluorinating a polymeric article in the presence of an electrical discharge (e.g., an alternating current corona discharge at atmospheric pressure) to produce a fluorinated article. In one embodiment, the method includes fluorinating the polymeric article in an atmosphere that includes fluorine containing species selected from the group consisting of elemental fluorine, fluorocarbons, hydrofluorocarbons, fluorinated sulfur, fluorinated nitrogen and combinations thereof. Examples of suitable fluorine containing species include C_5F_{12}, C_2F_6, CF_4, hexafluoropropylene, SF_6, NF_3, and combinations thereof.

In other embodiments, the method includes fluorinating the polymeric article in an atmosphere that includes elemental fluorine.

In other embodiments, the method of making the electret includes: (A) fluorinating a nonwoven polymeric fibrous web (i) in an atmosphere that includes fluorine containing species and an inert gas, and (ii) in the presence of an electrical discharge to produce a web having surface fluorination; and (B) charging the fluorinated web in a manner sufficient to produce an electret.

In other aspects, the invention features a method of filtering that includes passing an aerosol through the above-described electrets to remove contaminants.

The fluorinated electrets of the invention exhibit a relatively high oily mist resistance relative to non-fluorinated electrets.

GLOSSARY

In reference to the invention, these terms having the meanings set forth below: "electret" means a dielectric material exhibiting a quasi-permanent electrical charge. The term "quasi-permanent" means that the time constants characteristic for the decay of the charge are much longer than the time period over which the electret is used;

"surface modified" means that the chemical structure at the surface has been altered from its original state.

"surface fluorination" means the presence of fluorine atoms on a surface (e.g., the surface of an article);

"fluorine containing species" means molecules and moieties containing fluorine atoms including, e.g., fluorine atoms, elemental fluorine, and fluorine containing radicals;

"fluorinating" means placing fluorine atoms on the surface of an article by transferring fluorine containing species from a gaseous phase to the article by chemical reaction, sorption, condensation, or other suitable means;

"aerosol" means a gas that contains suspended particles in solid or liquid form; and

"contaminants" means particles and/or other substances that generally may not be considered to be particles (e.g., organic vapors).

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a plot of % DOP Penetration vs. DOP Load for Examples 36 and 37.

FIG. 2 is a plot of % DOP Penetration vs. DOP Load for Examples 38 and 39.

FIG. 3 is a plot of % DOP Penetration vs. DOP Load for Example 40.

DESCRIPTION OF PREFERRED EMBODIMENTS

The electret includes a surface modified polymeric article (e.g., a nonwoven polymeric fibrous web) produced by fluorinating a polymeric article. The electrets preferably have sufficient surface fluorination to provide oily mist resistance. One measure of oily mist resistance is how well the electret maintains its Quality Factor during challenge with an aerosol. The Quality Factor can be calculated from results obtained from the dioctylphthalate ("DOP") initial penetration test ("the DOP test"). The DOP test also provides a relative measure of the charge state of the filter. The DOP test procedure involves forcing DOP aerosol at a face velocity of 6.9 cm/second for a period of about 30 seconds through the sample, measuring the pressure drop across the sample (Pressure Drop measured in mmH_2O) with a differential manometer, and measuring the percent DOP penetration (DOPPen %). The Quality Factor (QF) (measured in $1/mmH_2O$) can be calculated from these values according to the following formula:

$$QF[1/mmH_2O] = \frac{-\mathrm{Ln}\frac{DOPPenetration(\%)}{100}}{PressureDrop\ [mmH_2O]}$$

The higher the Quality Factor at a given flow rate, the better the filtering performance of the electret.

Preferred electrets have a Quality Factor of at least about $0.25/mmH_2O$, preferably at least about $0.5/mmH_2O$, more preferably at least about $1.0/mmH_2O$.

Electron spectroscopy for chemical analysis ("ESCA") (also known as X-ray photoelectron spectroscopy ("XPS")) provides one measure of surface fluorination. Preferably the surface of the electret exhibits at least about 45 atomic % fluorine, more preferably at least about 50 atomic % fluorine when analyzed by ESCA. ESCA analyzes the elemental composition of the outermost surface (i.e., approximately 10 to 50 Å) of a specimen. ESCA can be used to detect all elements in the periodic table except helium and hydrogen.

The electret also has a CF_3:CF_2 ratio at the surface of the electret of at least about 0.25, preferably at least about 0.45, and more preferably greater than 0.9, as determined according to the Method For Determining CF_3:CF_2 ratio set forth in the Example section below.

In one embodiment, the electrets include nonwoven polymeric fibrous webs that include fibers such as, e.g., meltblown microfibers, staple fibers, fibrillated films, and combinations thereof The fibers can be formed from resins. Preferably the resin is a thermoplastic nonconductive, i.e., having a resistivity of greater than 10^{14} ohm-cm, resin. The resin used to form the fibers should be substantially free of materials such as antistatic agents that could increase the electrical conductivity or otherwise interfere with the ability of the fibers to accept and hold electrostatic charges.

Examples of useful thermoplastic resins include polyolefins such as, e.g., polypropylene, polyethylene, poly-(4-methyl-1-pentene), and combinations thereof, halogenated vinyl polymers (e.g., polyvinyl chloride), polystyrene, polycarbonates, polyesters, and combinations thereof.

Additives can be blended with the resin including, e.g., pigment, UV stabilizers, antioxidants, and combinations thereof.

Meltblown microfibers can be prepared as described in Wente, Van A., "Superfine Thermoplastic Fibers," *Industrial Eng. Chemistry*, Vol. 48, pp. 1342–1346 and in Report No. 4364 of the Naval Research laboratories, published May 25, 1954, entitled, "Manufacture of Super Fine Organic Fibers," by Wente et al. Meltblown microfibers preferably have an effective fiber diameter in the range of less than 1 to 50 μm as calculated according to the method set forth in Davies, C. N., "The Separation of Airborne Dust and Particles," Institution of Mechanical Engineers, London, Proceedings 1B, 1952.

The presence of staple fibers provides a more lofty, less dense web than a web constructed solely of meltblown microfibers. Preferably the electret contains more than 70% by weight staple fibers. Webs containing staple fibers are disclosed in U.S. Pat. No. 4,118,531 (Hauser).

Electrets that include a nonwoven polymeric fibrous web preferably have a basis weight in the range of about 10 to 500 g/m^2, more preferably about 10 to 100 g/m^2. The thickness of the nonwoven polymeric fibrous web is preferably about 0.25 to 20 mm, more preferably about 0.5 to 2 mm.

The nonwoven polymeric webs of the electret can also include particulate matter as disclosed, for example, in U.S. Pat. Nos. 3,971,373, (Braun), 4,100,324 (Anderson), and 4,429,001 (Kolpin et al.).

Electret Preparation

The electrets can be prepared by fluorinating a polymeric article, optionally in the presence of a surface modifying electrical discharge, and charging the fluorinated article to produce an electret.

The fluorination process includes modifying the surface of the polymeric article to contain fluorine atoms by exposing the polymeric article to an atmosphere that includes

fluorine containing species. The fluorination process can be performed at atmospheric pressure or under reduced pressure. The fluorination process is preferably performed in a controlled atmosphere to prevent contaminants from interfering with the addition of fluorine atoms to the surface of the article. The atmosphere should be substantially free of oxygen and other contaminants. Preferably the atmosphere contains less than 0.1% oxygen.

The fluorine containing species present in the atmosphere can be derived from fluorinated compounds that are gases at room temperature, become gases when heated, or are capable of being vaporized. Examples of useful sources of fluorine containing species include, fluorine atoms, elemental fluorine, fluorocarbons (e.g., C_5F_{12}, C_2F_6, CF_4, and hexafluoropropylene), hydrofluorocarbons (e.g., CF_3H), fluorinated sulfur (e.g., SF_6), fluorinated nitrogen (e.g., NF_3), fluorochemicals such as e.g., CF_3CF_3 and fluorochemicals available under the trade designation Fluorinert such as, e.g., Fluorinert FC-43 (commercially available from Minnesota Mining and Manufacturing Company, Minnesota), and combinations thereof.

The atmosphere of fluorine containing species can also include an inert diluent gas such as, e.g., helium, argon, nitrogen, and combinations thereof.

The electrical discharge applied during the fluorination process is capable of modifying the surface chemistry of the polymeric article when applied in the presence of a source of fluorine containing species, The electrical discharge is in the form of plasma, e.g., glow discharge plasma, corona plasma, silent discharge plasma (also referred to as dielectric barrier discharge plasma and alternating current ("AC") corona discharge), and hybrid plasma, e.g., glow discharge plasma at atmospheric pressure, and pseudo glow discharge. Preferably the plasma is an AC corona discharge plasma at atmospheric pressure. Examples of useful surface modifying electrical discharge processes are described in U.S. Pat. Nos. 5,244,780, 4,828,871, and 4,844 979.

Another fluorination process includes immersing a polymeric article into a liquid that is inert with respect to elemental fluorine, and bubbling elemental fluorine gas through the liquid to produce a surface fluorinated article. Examples of useful liquids that are inert with respect to fluorine include perhalogenated liquids, e.g., perfluorinated liquids such as Performance Fluid PF 5052 (commercially available from Minnesota Mining and Manufacturing Company). The elemental fluorine containing gas that is bubbled through the liquid can include an inert gas such as, e.g., nitrogen, argon, helium, and combinations thereof.

Charging the polymeric article to produce an electret can be accomplished using a variety of techniques, including, e.g., hydrocharging, i.e., contacting an article with water in a manner sufficient to impart a charge to the article, followed by drying the article, and DC corona charging. The charging process can be applied to one or more surfaces of the article.

One example of a useful hydrocharging process includes impinging jets of water or a stream of water droplets onto the article at a pressure and for a period sufficient to impart a filtration enhancing electret charge to the web, and then drying the article. The pressure necessary to optimize the filtration enhancing electret charge imparted to the article will vary depending on the type of sprayer used, the type of polymer from which the article is formed, the type and concentration of additives to the polymer, and the thickness and density of the article. Pressures in the range of about 10 to about 500 psi (69 to 3450 kPa) are suitable. An example of a suitable method of hydrocharging is described in U.S. Pat. No. 5,496,507 (Angadjivand et al.).

The jets of water or stream of water droplets can be provided by any suitable spray device. One example of a useful spray device is the apparatus used for hydraulically entangling fibers.

Examples of suitable DC corona discharge processes are described in U.S. Pat. No. Re. 30,782 (van Turnhout), U.S. Pat. No. Re. 31,285 (van Turnhout), U.S. Pat. No. Re. 32,171 (van Turnhout), U.S. Pat. No. 4,375,718 (Wadsworth et al.), U.S. Pat. No. 5,401,446 (Wadsworth et al.), U.S. Pat. No. 4,588,537 (Klasse et al.), and U.S. Pat. No. 4,592,815 (Nakao).

The fluorinated electrets formed by the methods described herein are suitable for use as, e.g., electrostatic elements in electro-acoustic devices such as microphones, headphones and speakers, fluid filters, dust particle control devices in, e.g., high voltage electrostatic generators, electrostatic recorders, respirators (e.g., prefilters, canisters and replaceable cartridges), heating, ventilation, air conditioning, and face masks.

The invention will now be described further by way of the following examples.

EXAMPLES

Test Procedures

Test procedures used in the examples include the following.

Method for Determining CF_3:CF_2

ESCA data was collected on a PHI 5100 ESCA system (Physical Electronics, Eden Prairie, Minn.) using a non-monochromatic MgKα x-ray source and a 45 degree electron takeoff angle with respect to the surface. The carbon (1s) spectra were peak fit using a nonlinear least-squares routine supplied by PHI (Physical Electronics, Eden Prairie, Minn.). This routine used a linear background subtraction, and a gaussian peak shape for the component peaks. The spectra were referenced to the hydrocarbon peak at 285.0 eV. The CF_3 and CF_2 components were identified as the peaks located at about 294 eV and 292 eV respectively (according to the procedure described in Strobel et al., J. Polymer Sci. A: Polymer Chemistry, Vol. 25, pp.1295–1307 (1987)). The CF_3:CF_2 ratio represent the ratio of the peak areas of the CF_3 and CF_2 components.

Initial Dioctylphthalate Penetration (DOP) and Pressure Drop Test Procedure

Initial DOP penetration is determined by forcing 0.3 micrometer diameter dioctyl phthalate (DOP) particles at a concentration of between 70 and 140 mg/m^3 (generated using a TSI No. 212 sprayer with four orifices and 30 psi clean air) through a sample of filter media which is 4.5 inches in diameter at a rate of 42.5 L/min (a face velocity of 6.9 centimeters per second). The sample is exposed to the DOP aerosol for 30 seconds until the readings stabilize. The penetration is measured with an optical scattering chamber, Percent Penetration Meter Model TPA-8F available from Air Techniques Inc.

Pressure drop across the sample is measured at a flow rate of 42.5 L/min (a face velocity of 6.9 cm/sec) using an electronic manometer. Pressure drop is reported in mm of water ("mm H_2O").

DOP penetration and pressure drop are used to calculate the quality factor "QF" from the natural log (ln) of the DOP penetration by the following formula:

$$QF[1/mmH_2O] = \frac{-Ln\frac{DOP\,Penetration(\%)}{100}}{Pressure\,Drop\,[mmH_2O]}$$

A higher initial QF indicates better initial filtration performance. A decreased QF effectively correlates with decreased filtration performance.

DOP Loading Test

DOP loading is determined using the same test equipment used in the DOP penetration and pressure drop tests. The test sample is weighed and then exposed to the DOP aerosol for at least 45 min to provide a minimum exposure of at least about 130 mg. DOP penetration and pressure drop are measured throughout the test at least as frequently as once per minute. The mass of DOP collected is calculated for each measurement interval from the measured penetration, mass of the filter web, and total mass of DOP collected on the filter web during exposure ("DOP Load").

Corona Fluorination

Example 1

A blown polypropylene microfiber web prepared from Exxon 3505G polypropylene resin (Exxon Corp.) and having an effective fiber diameter of 7.5 μm and a basis weight of 62 g/m^2 was prepared as described in Wente, Van A., "Superfine Thermoplastic Fibers," *Industrial Eng. Chemistry*, Vol. 48, pp. 1342–1346.

The blown microfiber web was then AC corona fluorinated in a 1% by volume C_2F_6 in helium atmosphere at a corona energy of 34 J/cm^2, which corresponded to a corona power of 2000W at a substrate speed of 1 m/min. The AC corona fluorination treatment was performed in an AC corona system that included the so-called "double-dielectric" electrode configuration with a ground roll consisting of 40 cm diameter nickel-plated aluminum roll covered with 1.5 mm of poly(ethylene terephthalate) and maintained at a temperature of 23° C. using recirculating, pressurized water. The powered electrodes consisted of 15 individual ceramic-covered electrodes (available from Sherman treaters Ltd., Thame, United Kingdom) each with a 15 mm square cross-section and an active length of 35 cm. The electrodes were connected to a model RS48-B (4 kW) variable-frequency power supply (available from ENI Power Systems Inc., Rochester, N.Y.). The net power dissipated in the AC corona was measured with a directional power meter incorporated into the ENI supply. The frequency of the output power was manually adjusted to about 16 kHz to obtain optimal impedance matching (minimum reflected power).

The AC corona system was enclosed within a controlled environment. Prior to treatment, the atmosphere surrounding the AC corona treatment system was purged with helium, and then continually flushed with 100 liters/min of 1% by volume C_2F_6 in helium, which was introduced near the electrodes.

The microfiber web was taped onto a carrier film of 0.05 mm thick bi-axially-oriented polypropylene (BOPP), and then placed on the ground roll such that the carrier film was in contact with the ground roll, causing one side of the blown microfiber web to be exposed to the discharge. After treatment, the blown microfiber web was flipped over, retaped to the carrier film, and AC corona treated a second time under the same conditions as the first treatment to expose the other side of the blown microfiber web to the discharge.

Example 2

A G100 Filtrete fibrillated film web (available from Minnesota Mining and Manufacturing), having a basis weight of 100 g/m^2, was corona fluorinated following the method described in Example 1, with the exception that the ground roll was maintained at a temperature of 25° C.

Example 3

A polyethylene meltblown microfiber web, prepared from Aspun PE-6806 polyethylene resin (DOW Chemical Company, Michigan) and having a basis weight of 107 g/m^2, was corona fluorinated following the method described in Example 2.

Example 4

A polyester staple fiber web (available from Rogers Corporation), having a basis weight of 200 g/m^2, was corona fluorinated following the method described in Example 2.

Example 5

A poly-4-methyl-1-pentene meltblown microfiber web prepared from TPX MX-007 poly-4methyl-1-pentene resin (Mitsui), and having a basis weight of 50 g/m^2 and an effective fiber diameter of 8.1 μm, was corona fluorinated following the method described in Example 2.

Examples 6–9

Examples 6–9 were prepared following the procedure in Example 1 except that the source of containing species was as follows: 1% CF_4 (Example 6), and 0.1% hexafluoropropylene (Example 7), 0.1% C_5F_{12} (Example 8), and 1.0% C_5F_{12} (Example 9).

The surface chemistry of each of the sample webs of Examples 1–9 was determined by ESCA analysis using a PHI 5100 ESCA system. The CF_3:CF_2 ratio was determined for each of the samples of Examples 1–9 from the ESCA data according to the above-described method. The results are reported in atomic % in Table I.

TABLE I

Example	Carbon	Nitrogen	Oxygen	Fluorine	CF_3:CF_2
1	43		5.7	51	1.09
2	44		6.2	50	1.37
3	49	0.2	8.2	42	1.10
4	42	0.5	7.8	49	0.99
5	44	0.0	2.9	53	1.19
6	41		3.5	55	0.86
7	41		2.7	56	0.97
8	42		6.4	52	0.91
9	43		5.2	51	0.89

Hydrocharging

Example 10

A fluorinated polypropylene blown microfiber web prepared as described above in Example 1, was passed over a vacuum slot at a rate of 5 cm/sec (centimeters/second) while deionized water was sprayed onto the web at a hydrostatic pressure of about 90 psi from a pair of Spraying Systems Teejet 9501 sprayer nozzles mounted 10 cm apart and centered 7 cm above the vacuum slot. The sample was then inverted and passed through the deionized water spray a second time such that both sides of the web were sprayed

with water. The deionized water spray was then removed and the web was again passed over the vacuum slot to remove excess water. The web was then hung to dry at ambient conditions.

Example 11

A fluorinated poly-4-methyl-1-pentene meltblown microfiber web prepared according to Example 5 was charged following the procedure of Example 10.

Examples 10A–11A

Examples 10A–11A were prepared following the procedures of Example 10 and 11 respectively, with the exception that, after corona fluorination and prior to hydrocharging, each of the fluorinated webs of Examples 10A–11A were subjected to an anneal at 140° C. (300° F.) for about 10 minutes.

Examples 13, 15, 16, 18 and 20

Examples 13, 15, 16, 18 and 20 were charged following the procedure of Example 10, with the exception that the fluorinated polymeric fibrous webs used in each of Examples 13, 15, 16, 18 and 20 were as follows: a fluorinated polyethylene microfiber web prepared according to Example 3 above (Example 13); a fluorinated polyester staple fiber web prepared according to Example 4 (Example 15); a fluorinated G100 Filtrete fibrillated film web prepared according to Example 2 (Example 16); a fluorinated polypropylene needle punched web (12 denier/fiber fibers of Exxon 3505 polypropylene resin), having a basis weight of about 200 g/m^2, and having been corona fluorinated following the method described in Example 1 (Example 18); and a polypropylene melt blown fine fiber web, having a basis weight of 46 g/m^2 and an effective fiber diameter of 3.7 μm, and having been corona fluorinated following the method described in Example 1 with the exception that 0.2% C_5F_{12} was used instead of 1% C_2F_6 (Example 20).

DC Corona Charging

Example 12

The fluorinated polyethylene meltblown microfiber web of Example 3 was charged using a DC corona discharge as follows. The fluorinated web was placed in contact with an aluminum ground plane, and then passed under an electrically positive DC corona source, in air, at a rate of about 1.2 meters/min, while maintaining a current to ground plane of about 0.01 mA/cm of corona source length. The distance from corona source to ground was about 4 cm.

Examples 14, 17, 19

Examples 14, 17 and 19 were charged following the procedure of Example 12, with the exception that the fluorinated polymeric fibrous webs for each of Examples 14, 17 and 19 were as follows: a fluorinated polyester staple fiber web prepared following the procedure of Example 4 (Example 14); a fluorinated polypropylene needle punched web (12 denier/fiber fibers made from Exxon 3505 polypropylene resin), having a basis weight of about 200 g/m^2, and having been corona fluorinated following the method described in Example 1 (Example 17); and a fluorinated polypropylene meltblown fine fiber web, having a basis weight of 46 g/m^2 and an effective fiber diameter of 3.7 μm, and having been corona fluorinated following the method described in Example 1 with the exception that 0.2% C_5F_{12} was used instead of 1% C_2F_6 (Example 19).

Examples 21–35

Examples 21–35 were prepared by fluorinating polypropylene blown micro fiber webs following the procedure of Example 1, with the exception that the source of fluorine for each of Examples 21–35 was as follows: 1% CF_4 (Examples 21–23), 1% C_2F_6 (Examples 24–26), 0.1% hexafluoropropylene (Examples 27–29), 0.1% C_5F_{12} (Examples 30–32), and 1.0% C_5F_{12} (Examples 33–35).

The fluorinated webs of Examples 23, 26, 29, 32, and 35 were then charged following the hydrocharging process described above in Example 10.

The fluorinated webs of Examples 22, 25, 28, 31 and 34 were then charged following the DC corona charging process described above in Example 12.

% DOP penetration ("% DOP PEN"), Pressure Drop (mmH_2O), and the Quality Factor ("QF") for each of the electrets of Examples 10–35 were determined according to the above-described Initial DOP Penetration and Pressure Drop Test Procedure. The results are summarized in Table II.

TABLE II

EXAMPLE	% DOP PEN	PRESSURE DROP	QF
10	0.119	3.65	1.84
10A	0.140	3.21	2.05
11	2.45	1.46	2.54
11A	0.778	1.60	3.04
12	56.1	1.14	0.51
13	38.1	1.15	0.84
14	78.3	0.38	0.64
15	65.6	0.41	1.03
16	27.3	0.40	3.25
17	70.4	0.19	1.85
18	37.6	0.19	5.15
19	0.81	10.58	0.46
20	0.006	11.3	0.86
21	55.6	2.83	0.21
22	15.0	3.28	0.58
23	0.288	3.09	1.89
24	54.1	3.05	0.20
25	14.3	3.32	0.59
26	0.243	3.08	1.95
27	59.0	2.81	0.19
28	16.2	2.80	0.65
29	0.276	2.90	2.03
30	52.5	3.15	0.20
31	14.0	3.11	0.63
32	0.250	2.99	2.00
33	45.3	3.10	0.26
34	14.9	2.93	0.65
35	0.244	3.14	1.92

Example 36–39

Four fluorinated, polypropylene microfiber webs were prepared according to Example 1 with the exception that the source of fluorine containing species was as follows: 0.1% hexafluoropropylene ("HFP") (Examples 36 and 38) and 0.1% $C5F_{12}$ (Example 37 and 39).

Examples 36 and 37 further included charging the fluorinated polypropylene webs following the hydrocharging charging procedure of Example 10.

Examples 38 and 39 further included charging the fluorinated polypropylene webs following the DC corona charging procedure of Example 12.

Examples 36–39 were subjected to the above-described DOP Loading Test. The % DOP Penetration versus DOP loading (the amount of DOP collected on the web in grams) for each of Examples 36–39 was measured according to the above-described DOP Loading Test Procedure. The result-

ing data are plotted as % DOP penetration versus DOP load (grams) in FIGS. 1 and 2 as follows: Examples 36 and 37 (indicated with x's and solid circles respectively) (FIG. 1), and Examples 38 and 39 (indicated with x's and solid circles respectively) (FIG. 2).

Example 40

A 7 in. by 7 in. sample of polypropylene microfiber web having a basis weight of 61 g/m^2 was placed under a nitrogen atmosphere. A gaseous mixture of 5% by volume elemental fluorine diluted in nitrogen was passed through the polypropylene microfiber web at a rate of 1.0 l/min for 10 minutes. The fluorine concentration was then increased to 10% by volume diluted in nitrogen and passed through the web at a rate of 1.0 l/min for an additional 20 minutes.

The sample was then analyzed by ESCA and determined to have 62 atomic % fluorine and a CF_3:CF_2 ratio of 0.59, as determined according to the above-described Method for Determining CF_3:CF_2.

The sample was then charged using a DC corona discharge as described above in Example 12, and subjected to the above-described DOP Loading Test. The resulting data are plotted as % DOP Penetration versus DOP Load (grams) in FIG. 3.

Example 41

A polypropylene blown microfiber web, having a basis weight of 20 g/m^2 and a web width of 15 cm, was vacuum glow-discharge treated in a C_5F_{12} environment. The glow-discharge treatment was performed in a vacuum chamber. The vacuum chamber contained a roll-to-roll glow discharge system consisting of an unwind roller, glow discharge electrodes, and a windup roller for the continuous treatment of the blown microfiber web. Two stainless steel electrodes were in the parallel plate configuration, each electrode was 20 cm wide and 33 cm long and they were separated by a gap of 2.5 cm. The top electrode was grounded and the bottom electrode was powered by a 13.56 MHz rf generator (Plasma-Therm). The web traveled between the two electrodes and in contact with the top, grounded electrode so that one side of the web was exposed to the discharge.

After loading the roll of blown microfiber web onto the unwind roller under C_5F_{12} vapor at a pressure of 0.1 Torr. The blown microfiber web was advanced through the electrodes at a speed of 17 cm/min to achieve an exposure time to the plasma of 2 minutes. The discharge power was 50W. After the first side was treated, the chamber was vented and the web roll replaced onto the unwind roller to allow the other side of the web to be treated. The treatment of the second side of the web occurred under the same conditions as the first side. After the fluorination, Example 41 was DC-corona charged following the process described above in Example 12.

% DOP Penetration ("% DOP PEN") for Example 41 was determined according to the above-described Initial DOP Penetration and Pressure Drop Test Procedure. The results are summarized in Table III.

TABLE III

Loading Time (min)	% DOP Penetration Example 14
0.5	28
10	28

Other embodiments are within the following claims. Although the electret has been described with reference to nonwoven polymeric fibrous webs, the electret can be a variety of polymeric articles including, e.g., those polymeric articles described in U.S. patent application Ser. No. 09/106,506, entitled, "Structured Surface Filter Media," (Insley et al.), filed on Jun. 18, 1998.

All of the patents and patent applications cited above are incorporated by reference into this document in total.

What is claimed is:

1. A method of filtering contaminants, said method comprising:

passing an aerosol through a plasma surface modified nonwoven polymeric web electret to remove contaminants from the aerosol,

the nonwoven polymeric web comprising plasma surface fluorination, the electret, when tested according to the Initial DOP Penetration Test and the DOP Loading Test prior to contact with the aerosol, exhibiting a DOP penetration of less than 20% for a DOP load from 0.05 grams to 0.2 grams.

2. The method of claim 1 wherein the electret is part of a dust particle control device.

3. The method of claim 1, wherein the electret exhibits a DOP penetration of less than 15% for a DOP load of from 0.05 grams to 0.2 grams prior to contact with the aerosol.

4. The method of claim 1, wherein the electret exhibits a DOP penetration of less than 10% for a DOP load from 0.05 grams to 0.2 grams prior to contact with the aerosol.

5. The method of claim 1, wherein the electret exhibits a DOP penetration of no greater than 5% for a DOP load from 0.05 grams to 0.2 grams prior to contact with the aerosol.

6. The method of claim 1, wherein the electret exhibits a DOP penetration of less than 10% for a DOP load of from 0.02 grams to 0.08 grams prior to contact with the aerosol.

7. The method of claim 1, wherein the web comprises at least 45 atomic % fluorine as detected by ESCA.

8. The method of claim 1, wherein the web comprises a CF_3:CF_2 ratio of at least 0.45 as determined according to the Method of Determining CF_3:CF_2.

9. The method of claim 1, wherein the web comprises at least 45 atomic % fluorine as detected by ESCA and a CF_3:CF_2 ratio of at least 0.45 as determined according to the Method of Determining CF_3:CF_2.

10. The method of claim 1, wherein the web comprises a surface fluorination of at least 50 atomic % fluorine as detected by ESCA.

11. The method of claim 1, wherein the web comprises at least 50 atomic % fluorine as detected by ESCA and a CF_3:CF_2 ratio of at least 0.25 as determined according to the Method for Determining CF_3:CF_2.

12. The method of claim 1, wherein the web comprises at least 50 atomic % fluorine as detected by ESCA and a CF_3:CF_2 ratio of at least 0.45 as determined according to the Method for Determining CF_3:CF_2.

13. The method of claim 1, wherein the web comprises a CF_3:CF_2 ratio of at least 0.9.

14. The method of claim 1, wherein the web comprises at least 50 atomic % fluorine as detected by ESCA and a

CF_3:CF_2 ratio of at least 0.9 as determined according to the Method for Determining CF_3:CF_2.

15. The method of claim 1, wherein the electret exhibits a Quality Factor of at least 0.5/mmH_2O prior to contact with the aerosol.

16. The method of claim 1, wherein the electret exhibits a Quality Factor of at least 2/mmH_2O prior to contact with the aerosol.

17. The method of claim 1, wherein the web comprises fibers selected from the group consisting of polycarbonate, polyolefin, polyester, halogenated polyvinyl, polystyrene, or a combination thereof.

18. The method of claim 1, wherein the web comprises fibers selected from the group consisting of polypropylene, poly-(4-methyl-1-pentene), or a combination thereof.

19. The method of claim 1, wherein the web comprises meltblown microfibers.

20. The method of claim 19, wherein the microfibers comprise polypropylene.

21. The method of claim 1, wherein the web has a basis weight of from 10 to 100 g/m^2.

22. A method of filtering contaminants, said method comprising:

passing an aerosol through an electret to remove contaminants from the aerosol,

the electret comprising a nonwoven polymeric web comprising at least about 45 atomic % fluorine as detected by ESCA and a CF_3:CF_2 ratio of at least about 0.45 as determined according to the Method of Determining CF_3:CF_2,

the electret having a Quality Factor of at least about 0.25/mmH_2O prior to contact with the aerosol.

23. The method of claim 22, wherein the electret has a Quality Factor of at least about 0.5/mmH_2O prior to contact with the aerosol.

24. The method of claim 22, wherein the electret has a Quality Factor of at least about 1/mmH_2O prior to contact with the aerosol.

25. The method of claim 22, wherein the nonwoven polymeric web comprises fibers selected from the group consisting of polycarbonate, polyolefin, polyester, halogenated polyvinyl, polystyrene, or a combination thereof.

26. The method of claim 22, wherein the nonwoven polymeric web comprises fibers selected from the group consisting of polypropylene, poly-(4-methyl-1-pentene), or a combination thereof.

27. The method of claim 22, wherein the nonwoven polymeric web comprises meltblown microfibers.

28. A method of filtering contaminants, said method comprising:

passing an aerosol through an electret to remove contaminants from the aerosol,

the electret comprising a nonwoven polymeric web comprising at least about 50 atomic % fluorine as detected by ESCA and a CF_3:CF_2 ratio of at least about 0.25 as determined according to the Method for Determining CF_3:CF_2,

the electret having a quality factor of at least about 0.25/mmH_2O prior to contact with the aerosol.

29. A method of filtering contaminants, said method comprising:

passing an aerosol through an electret to remove contaminants from the aerosol,

the electret comprising a nonwoven polymeric web comprising microfibers, the web having surface fluorination that comprises CF_3 and CF_2 at a CF_3:CF_2 ratio of at least 0.45 as determined according to the Method for Determining CF_3:CF_2,

the electret having a quality factor of at least about 0.25/mmH_2O prior to contact with the aerosol.

30. The method of claim 29, wherein the electret has a Quality Factor of at least about 1.0/mmH_2O.

31. The method of claim 29, wherein the web has a surface fluorination of at least about 45 atomic % fluorine as detected by ESCA.

32. The method of claim 29, wherein the CF_3:CF_2 ratio is at least 0.9.

33. The method of claim 29, wherein the microfibers are melt-blown microfibers that have an effective fiber diameter of 1 to 50 μm.

34. The method of claim 29, wherein the microfibers comprise polyolefin.

35. The method of claim 29, wherein the microfibers comprise polypropylene.

36. The method of claim 29, wherein the nonwoven web has a basis weight of 10 to 100 g/m^2.

37. The method of claim 29, wherein the nonwoven web has a thickness of 0.25 to 20 mm.

* * * * *

UNITED STATES PATENT AND TRADEMARK OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 6,808,551 B2
DATED : October 26, 2004
INVENTOR(S) : Jones, Marvin E.

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

Column 4,
Line 18, after "thereof" insert -- . --.

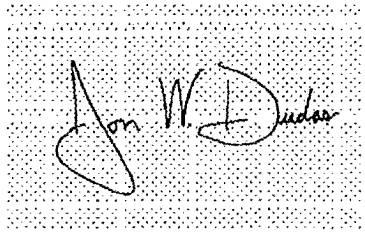
Column 5,
Line 17, delete "CF_3CF_3" and insert -- CF_3OCF_3 --, therefor.
Line 28, delete "," and insert -- . --, therefor.
Line 37, delete "4,844 979" and insert -- 4,844,979 --, therefor.

Column 6,
Line 66, delete "(In)" and insert -- (Ln) --, therefor.

Column 8,
Line 24, delete "poly-4methyl-1-pentene" and insert -- poly-4-methyl-1-pentene --, therefor.
Line 32, insert -- fluorine -- before "containing".

Signed and Sealed this

Fifteenth Day of February, 2005



JON W. DUDAS
Director of the United States Patent and Trademark Office

CERTIFICATE OF SERVICE

I hereby certify that, on this 3rd day of November, 2014, I filed the foregoing Brief for Appellants 3M Innovative Properties Company and 3M Company with the Clerk of the United States Court of Appeals for the Federal Circuit via the CM/ECF system, which will send notice of such filing to all registered CM/ECF users.

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CERTIFICATE OF COMPLIANCE

Pursuant to Fed. R. App. P. 32(a)(7)(C), the undersigned hereby certifies that this brief complies with the type-volume limitation of Fed. R. App. P. 32(a)(7)(B) and Circuit Rule 32(b).

1. Exclusive of the exempted portions of the brief, as provided in Fed. R. App. P. 32(a)(7)(B), the brief contains 13,996 words.

2. The brief has been prepared in proportionally spaced typeface using Microsoft Word 2010 in 14 point Times New Roman font. As permitted by Fed. R. App. P. 32(a)(7)(C), the undersigned has relied upon the word count feature of this word processing system in preparing this certificate.

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